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SUMMARY TECHNICAL REPORT
OF THE
NATIONAL DEFENSE RESEARCH COMMITTEE

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SUMMARY TECHNICAL REPORT OF DIVISION 11, NDRC

VOLUME 2

MISCELLANEOUS CHEMICAL ENGINEERING PROBLEMS

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NOTES ON THE ORGANIZATION OF NDRC

The duties of the National Defense Research Committee were (1) to recommend to the Director of OSRD suitable projects and research programs on the instrumentalities of warfare, together with contract facilities for carrying out these projects and programs, and (2) to administer the technical and scientific work of the contracts. More specifically, NDRC functioned by initiating research projects on requests from the Army or the Navy, or on requests from an allied government transmitted through the Liaison Office of OSRD, or on its own considered initiative as a result of the experience of its members. Proposals prepared by the Division, Panel, or Committee for research contracts for performance of the work involved in such projects were first reviewed by NDRC, and if approved, recommended to the Director of OSRD. Upon approval of a proposal by the Director, a contract permitting maximum flexibility of scientific effort was arranged. The business aspects of the contract, including such matters as materials, clearances, vouchers, patents, priorities, legal matters, and administration of patent matters were handled by the Executive Secretary of OSRD.

Originally NDRC administered its work through five divisions, each headed by one of the NDRC members. These were:

Division A — Armor and Ordnance
Division B — Bombs, Fuels, Gases, & Chemical Problems
Division C — Communication and Transportation
Division D — Detection, Controls, and Instruments
Division E — Patents and Inventions

In a reorganization in the fall of 1942, twenty-three administrative divisions, panels, or committees were created, each with a chief selected on the basis of his outstanding work in the particular field. The NDRC members then became a reviewing and advisory group to the Director of OSRD. The final organization was as follows:

Division 1 — Ballistic Research
Division 2 — Effects of Impact and Explosion
Division 3 — Rocket Ordnance
Division 4 — Ordnance Accessories
Division 5 — New Missiles
Division 6 — Sub-Surface Warfare
Division 7 — Fire Control
Division 8 — Explosives
Division 9 — Chemistry
Division 10 — Absorbents and Aerosols
Division 11 — Chemical Engineering
Division 12 — Transportation
Division 13 — Electrical Communication
Division 14 — Radar
Division 15 — Radio Coordination
Division 16 — Optics and Camouflage
Division 17 — Physics
Division 18 — War Metallurgy
Division 19 — Miscellaneous
Applied Mathematics Panel
Applied Psychology Panel
Committee on Propagation
Tropical Deterioration Administrative Committee

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NDRC FOREWORD

As events of the years preceding 1940 revealed more and more clearly the seriousness of the world situation, many scientists in this country came to realize the need of organizing scientific research for service in a national emergency. Recommendations which they made to the White House were given careful and sympathetic attention, and as a result the National Defense Research Committee [NDRC] was formed by Executive Order of the President in the summer of 1940. The members of NDRC, appointed by the President, were instructed to supplement the work of the Army and the Navy in the development of the instrumentalities of war. A year later, upon the establishment of the Office of Scientific Research and Development [OSRD], NDRC became one of its units.

The Summary Technical Report of NDRC is a conscientious effort on the part of NDRC to summarize and evaluate its work and to present it in a useful and permanent form. It comprises some seventy volumes broken into groups corresponding to the NDRC Divisions, Panels, and Committees.

The Summary Technical Report of each Division, Panel, or Committee is an integral survey of the work of that group. The first volume of each group's report contains a summary of the report, stating the problems presented and the philosophy of attacking them, and summarizing the results of the research, development, and training activities undertaken. Some volumes may be "state of the art" treatises covering subjects to which various research groups have contributed information. Others may contain descriptions of devices developed in the laboratories. A master index of all these divisional, panel, and committee reports which together constitute the Summary Technical Report of NDRC is contained in a separate volume, which also includes the index of a microfilm record of pertinent technical laboratory reports and reference material.

Some of the NDRC-sponsored researches which had been declassified by the end of 1945 were of sufficient popular interest that it was found desirable to report them in the form of monographs, such as the series on radar by Division 14 and the monograph on sampling inspection by the Applied Mathematics Panel. Since the material treated in them is not duplicated in the Summary Technical Report of NDRC, the monographs are an important part of the story of these aspects of NDRC research.

In contrast to the information on radar, which is of widespread interest and much of which is released to the public, the research on subsurface warfare is largely classified and is of general interest to a more restricted group. As a consequence, the report of Division 6 is found almost entirely in its Summary Technical Report, which runs to 23 volumes. The extent of the work of a division cannot therefore be judged solely by the number of volumes devoted to it in the Summary Technical Report of NDRC: account must be taken of the monographs and available reports published elsewhere.

One can claim on behalf of Division 11 that the results of its work contributed directly and dramatically to the successful prosecution and triumphant termination of World War II. It was Division 11, under the leadership first of R. P. Russell, then E. P. Stevenson, and later H. M. Chadwell, which developed the incendiary bombs with which Japan's industrial plants were reduced to ashes. Filled with jellied gasoline, the AN-M69 incendiary was credited with the highest efficiency of any bomb against Japanese factories and dwellings. More than 40,000 tons of AN-M69 bombs were dropped on Japanese cities.

Division 11 likewise applied the use of thickened fuels to portable and mechanized flame throwers, which were employed with great success against the enemy in the Pacific. Other sections of the Division did important work in developing improved techniques for the production of oxygen for military uses, and in solving numerous other problems in the field of chemical engineering, one of the most valuable contributions being the development of new hydraulic fluids.

This Summary Technical Report of Division 11, prepared under the direction of the Division Chief and authorized by him for publication, describes the activities of the Division and its contractors. It stands as a testimonial to the imagination and resourcefulness of American scientists and industrial engineers and as a record of wartime accomplishment worthy of grateful recognition.

Vannevar Bush, Director
Office of Scientific Research and Development

J. B. Conant, Chairman
National Defense Research Committee

FOREWORD

For administrative purposes and because of the diverse nature of the problems studied by Division 11 (Chemical Engineering) of NDRC, three independent sections were created: Section 11.1 (Oxygen Problems), Section 11.2 (Miscellaneous Chemical Engineering Problems), and Section 11.3 (Fire Warfare). The work of each of the three sections is presented in an individual volume of the Summary Technical Report of NDRC.

The work of Section 11.2, reported in this volume, was unusual in that it involved research and development projects of such extreme diversification. As the activities of NDRC increased, various research programs took shape and were defined by the creation of divisions and sections. Section 11.2 remained as the residue of this development of the NDRC organization, being assigned quite miscellaneous chemical and chemical engineering projects not logically falling in the field of effort of the other sections of Division 11, or of Divisions 8, 9, and 10. This may serve to explain the unusual variety of projects indicated by the list of chapter headings.

The work of Section 11.2 was carried out under the direction of Mr. R. P. Russell (January and February 1943), Mr. E. P. Stevenson (March 1943 to February 1945), and Dr. H. M. Chadwell (March 1945 to termination) as Chiefs of Division 11 for the periods indicated, and of Dr. T. K. Sherwood (January 1943 to termination) as Chief of Section 11.2. Assisting them were Mr. R. C. Wilcox, Mr. Abbott Byfield, and Mr. F. E. Vinal as Technical Aides of Section 11.2.

In 1942 the section was presented with several problems by the Office of the Quartermaster General, and certain contracts were initiated with research and development agencies. Mr. Stanley Lovell joined the section as a Technical Aide at that time, devoting his efforts entirely to the OQMG projects. In 1943 this work was transferred to a Committee on Quartermaster Problems of the National Research Council, with L. W. Bass as chairman, where it was handled by sub-contracts under a prime contract between OSRD and NRC. This contract was supervised directly by the office of the chairman of NDRC. It was returned to Section 11.2 in the late spring of 1944, however, and in December 1944, was transferred to the OQMG. Although started in Section 11.2, the work accomplished on the various projects for the Quartermaster General was done almost entirely under the direction of the NRC Committee on Quartermaster Problems, consisting of W. M. Clark, K. H. Condit, S. D. Kirkpatrick, N. A. Shepard, Raymond Stevens, and F. W. Willard, with L. W. Bass as chairman.

The Summary Technical Report of this section is divided in two parts: Part I, on general projects, prepared by T. K. Sherwood and R. C. Wilcox, with the assistance of several of the contractors; and Part II, covering the Quartermaster problems, prepared by W. G. Parks, Technical Aide to the NRC Committee on Quartermaster Problems. The coordination within the section was supervised first by R. C. Wilcox and later by D. Churchill, Jr. To all of these men the Division Chief wishes to express his sincere thanks.

In many instances industrial and university contractors undertook research of a definitely hazardous character without hesitation or complaint. While engaged in tests of an antisubmarine flare, Charles R. Hoover lost his life in a blimp accident in 1942. The courage and devotion of such men, though seldom publicized, were necessary parts of the successful whole war effort.

Because of the varied nature of the projects, it was necessary to obtain the advice and guidance of specialists in several fields, and acknowledgment is due a great many people in this regard. Special thanks are due G. H. B. Davis, C. K. Drinker, W. K. Lewis, A. J. Weith, and M. R. Fenske. The successful administration of the section's affairs was largely in the hands of R. C. Wilcox, Technical Aide, whose loyalty and good judgment made possible much of what was accomplished.

Acknowledgment is made of the valuable assistance rendered by the Services and by the liaison officers assigned by them to the various projects.

The Division Chief also wishes to acknowledge with thanks the valuable help and guidance in broad phases of the program and policy of Dr. Roger Adams, Member of NDRC.

H. M. Chadwell
Chief, Division 11
Thomas K. Sherwood
Chief, Section 11.2

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By *T. K. Sherwood* and *F. C. Wilcox*

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Chapter 1

HYDRAULIC FLUIDS

1.1 SUMMARY

THE VARIOUS MILITARY uses of hydraulic fluids and recoil oils are described, and the desirable and necessary characteristics of these fluids are summarized. Fluids available in 1941 proved to have many deficiencies, in many cases of such a serious nature as to make the gun turret or other mechanism inoperative at low temperatures. It was found that suitable fluids, having excellent properties at both low and high temperatures, could be prepared by incorporating high-polymeric additives such as certain acrylic esters in properly prepared hydrocarbon base stocks. In addition to the viscosity-temperature slope, many other factors such as shear stability, tackiness, and oxidation resistance proved to be important.

Specifications were issued for aircraft hydraulic fluids, Army recoil oils, and Navy shipboard hydraulic fluids, and large quantities of each were procured. As compared with the 1941 specification, the new aircraft fluid had a greater viscosity above 130 F and a viscosity of 500 or less in place of the previous 7,000 centistokes at −40 F. This reduction in low-temperature viscosity was sufficient to mean the difference between the equipment working and not working in many cases. A satisfactory noninflammable hydraulic fluid for general use was not developed.

1.2 INTRODUCTION

Hydraulic fluids are used in Army, Navy, and Air Force ordnance equipment for the transmission as well as the dissipation of energy over wide temperature ranges. A simple illustration of the application of hydraulic power is hydraulic brakes commonly employed on automobiles. In their operation a fluid transmits the power to the brake through a small tube. This is simpler and more effective than a relatively complex system of mechanical links and levers to accomplish the same result.

Hydraulically operated devices on aircraft are of many types. They include actuating equipment for landing gears, wing flaps, brakes, and control and steering equipment pertaining to the automatic pilot. In certain instances there are time sequences involved, such as in raising and lowering undercarriages and their doors or housings. On flying boats there are hydraulically operated retractable floats. Bomb doors are operated against varying aerodynamic loads. In some of the large planes, there is hydraulic steering on the ground through the nose wheel. Hydraulic fluids are necessary in shock absorbers. In addition to the foregoing, there are important applications in gun turrets and fire controls. In some of the very large airplanes it is necessary to apply hydraulic operation to the surface controls, as the forces involved in flight are so large that manual operation is no longer possible. Some variable-pitch propellers are also hydraulically operated. Combinations of hydraulic and electric devices are frequently employed.

Navy ordnance equipment employs hydraulic power to train and elevate the various guns ranging from the 40-mm to the large 16-in. size. Projectile and ammunition hoists are also hydraulically operated on shipboard. Ships are steered by the application of hydraulic power. On aircraft carriers hydraulic fluids are used in arresting gears and catapults, in addition to all the other uses normally employed on combat vessels.

In Army ordnance equipment, hydraulic fluids are used in recoil mechanisms of field guns, howitzers, and antiaircraft guns. In these cases part of the energy of recoil is absorbed by the oil and converted into heat, as the oil is forced through various throttling devices and orifices. Hydraulic equipment is also used to train guns and carry out other fire-control operations in tanks and armored equipment.

In addition to the foregoing, there are certain other highly specialized uses for specific fluids. Although the quantity involved here is not as large, the application is equally important. Compass oils are one example. Special damping fluids for fire-control equipment is another illustration of the use of specific liquids for highly specialized purposes in warfare.

1.3 CHARACTERISTICS OF DESIRABLE HYDRAULIC FLUIDS

The hydraulic fluids used in ordnance equipment must have sufficient viscosity and lubrication properties to provide adequate protection to all the moving parts at the highest temperatures encountered during use. These may exceed 250 F. Sufficient viscosity is also needed to prevent internal and external leakage. Many hydraulic devices will not function properly if there is excessive leakage past valves, pistons, and other close-fitting and moving parts within the hydraulic system. Leakage past glands, gaskets, and other sealing devices into the surroundings is obviously objectionable. At the same time such fluids must possess a relatively high fluidity at the lowest operating temperatures encountered in the different hydraulic systems, for it is obvious that a sluggish or immovable fluid can readily cause malfunctioning of hydraulic equipment. Hydraulic fluids should be nontoxic; they should not corrode the various metals in the hydraulic system and should not be harmful to the gaskets and sealing devices which are usually made of synthetic rubber. They must not oxidize or change chemically so as to impair their properties and usefulness. They should not evaporate readily, and their stability and service life should be long. In most instances it is also desirable that they offer some protection against rusting.

The mechanical design of the various hydraulic mechanisms and recoil devices in existence and in production in the early part of World War II was such as to require the use of a new hydraulic fluid if this equipment was to work over the wide temperature ranges anticipated, and if it was to remain in operation over long periods of time under the most adverse climatic and service conditions. One of the important requirements was to improve the viscosity-temperature properties of the fluid. This would enable much of the equipment to be utilized over temperature ranges not otherwise possible without making changes in its design or in its manufacture. It was obviously a simple procedure to remove the old oil and replace it with a new variety, if this would accomplish the desired results. In a large number of instances, this simple expedient of using an improved hydraulic oil has extended the climatic and service operability of ordnance equipment well beyond the requested ranges.

An ideal hydraulic fluid has a relatively large number of specific properties, and each must be held within definite limits. Since it was very doubtful that a single pure substance could possess all of these properties (which may be 20 or more), it was evident that this ideal fluid would have to be a carefully prepared mixture of various ingredients. However, the problem of retaining each property somewhere near its optimum value without jeopardizing any of the other properties, or the availability of the fluid, was a sizable task. Hundreds of combinations of materials were tested before the proper formula was found.

1.4 AIRCRAFT FLUIDS

A fluid with a much flatter viscosity-temperature curve than was available in the two specification fluids used at the start of World War II was desired for aircraft. Specification 3580 related to a hydrocarbon base fluid, while specification 3586 covered a castor oil base fluid. It was also desired to have only one type and grade of aircraft hydraulic fluid to eliminate confusion, and to simplify problems of manufacture and supply. In the early part of the war it was found that 3580 or 3586 fluids did not permit certain vitally important hydraulic devices on aircraft, such as flight equipment, turrets, and other fire-control devices to operate satisfactorily. To obtain relief, various expedients were used, such as adding kerosene to the hydrocarbon base hydraulic fluid then in use. In addition, some of the newer develop-

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ments in hydraulic mechanisms employed pressures up to 3,000 psi. A fluid was needed suitable for these pressures as well as for higher operating temperatures, which were frequently encountered when compact hydraulic units were in operation for long periods of time. There were also problems of leakage. Many of these could be solved if the fluid had less effect on the various rubber parts which were used in hydraulic systems for sealing purposes, and if it would not thin out too much at the higher temperatures. It was apparent that the new hydraulic oil should be fluid at −65 F, and that its viscosity should be as uniform as possible throughout the range from about −65 to 250 F.

1.5 RECOIL OILS

Improved Army ordnance fluids were desired in order to make the various recoil mechanisms on the different guns operative with a single fluid at temperatures ranging from about –30 to about 200 F. There were several grades of recoil oil in use at the start of the war, and it was desired to eliminate these and consolidate them into one grade. It was found that some of the recoil mechanisms would not operate satisfactorily at high temperatures if they had been provided originally with a light oil, which would enable their low-temperature performance to be satisfactory. If a fluid could be provided that would permit all the equipment to be operated at all temperatures to be encountered in the field, it would simplify maintenance as well as supply. Since hydraulic fluids are used to train and elevate guns, it was also desired to use this recoil oil as the hydraulic fluid for these operations.

1.6 HYDRAULIC FLUIDS FOR SHIPBOARD USE

Some of the problems relating to Army ordnance equipment were also prevalent in Navy equipment. For example, in connection with Navy antiaircraft guns, it was found that the existing fluid would not provide satisfactory control in elevation and in train at temperatures below about +30 F. There was no time to redesign these complex units. It was imperative to have a fluid with a flatter viscosity-temperature curve so that this vital equipment could operate at temperatures from subzero to the highest to be encountered, which might extend to 250 or 300 F. Difficulties were also arising because some of the fluids then in use did not have an adequate service life. Degradation of some of these fluids occurred in service. This was a very serious matter because of the complexity and relative inaccessibility of hydraulic equipment on shipboard. Protection against rusting was desired. In addition, improvements were also needed in the lubricating properties and in the overall stability and performance characteristics. While it was clear that the new hydraulic fluid should possess a flatter viscosity-temperature curve, it was also evident that this property alone could not solve all the difficulties. Consequently, a complete and thorough analysis was made of all requirements of Navy hydraulic fluids so that the necessary research and development work could proceed in a logical manner toward the attainment of improved composition and formula that would eliminate the troubles.

1.7 USE OF POLYMERIC ADDITIVES

One way to produce liquids having lower viscosity-temperature coefficients was to employ certain soluble linear-type polymers. In view of the practicability of this plan, it was given detailed study. In order to meet all other requirements it was necessary to investigate a very large number of chemical compounds and petroleum products. Even after investigating many hundred fluids, it was found that only 3 or 4 per cent of these showed promise of meeting most of the requirements. Considerably more work was required to find the best formulas using materials that could be converted promptly into hydraulic fluids. Nearly 200 different polymers and over 20 different petroleum base stocks were investigated. In addition, all new developments by the chemical industry, including silicone oils, chlorinated

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hydrocarbons, and the new fluids developed by the Carbide and Carbon Chemicals Company, were studied from the standpoint of fulfilling all specific Service needs, as well as meeting the requirements for production and availability. However, each of these new fluids was found to be deficient in some important property.

As the work progressed, it became clear that the best overall combination of properties could be obtained by using selected petroleum fractions along with carefully prepared oil-soluble polymers and other additives. This type of fluid had the additional advantage that it could be produced fairly readily with the minimum of new materials, chemicals, and plant facilities. This was very important at the period in World War II when manpower and supplies were extremely critical. Two linear-type polymeric compounds were found to be suitable for use in certain carefully selected petroleum fractions. One is polybutene, which is a special variety of polymerized isobutylene. The other is an acrylic acid ester polymer known as Acryloid. It was necessary to study a wide molecular weight range of these polymers to find the optimum molecular sizes for hydraulic fluids. In the case of the Acryloids, it was also essential to study the chemical make-up of these polymers in addition to their size, for this markedly affected the low-temperature properties of the fluids as well as their viscosity-temperature characteristics.

Acrylic acid ester polymers compounded from alcohols containing more than about 12 carbon atoms formed thixotropic gels at temperatures below −30 F. Polymers made from alcohols having less than about 4 carbon atoms, or aryl substituted alcohols, possessed limited solubility in conventional hydrocarbon base stocks. Acryloid polymers formulated from the intervening alcohols (about 4 to 12 carbon atoms) were generally found to be suitable for use in petroleum base stocks. Studies were then carried out to determine which molecular types were the most suitable for improving the viscosity-temperature characteristics of hydraulic fluids. The use of certain alcohols was also limited by their commercial availability. The 4-, 8-, 10-, and 12-carbon atom alcohols varied in their availability. During the early portion of the work the 8-carbon atom alcohol was more available than the others. When this alcohol became difficult to obtain it was found possible to prepare polymers by using the other alcohols. Mixtures of the 8- and 10-carbon atom alcohols, and the 8-, 10-, and 12-carbon atom alcohols were found to be suitable for preparing polymers when the supply of any one alcohol was limited. Little basic difference was found between polymers compounded from one alcohol and those compounded from alcohol mixtures within the 8- to 12-carbon atom range. Acrylic acid ester polymers, prepared from mixtures of the 4- and 12-carbon atom alcohols, were also found to be suitable for improving the viscosity-temperature characteristics of a petroleum base stock.

The molecular weight of the polymers was found to be of utmost importance. For an Acryloid polymer prepared from a given alcohol or alcohol mixture, the molecular weight was found to have an important effect on the viscosity-temperature properties of any base stock in which it was employed. Because of the difficulty of measuring the actual molecular weights of the polymers, a relative molecular weight scale was devised which was found to be applicable to the range of polymer molecular weights employed in the formulation of hydraulic fluids. This scale consisted of measuring the viscosity in centistokes at 210 F of a 30 (weight) per cent solution of the active Acryloid polymer in a standard hydrocarbon base stock of essentially reproducible viscosity. In order to judge the ability of a polymeric additive to improve the viscosity-temperature characteristics of a hydraulic fluid, a quantity of the active polymeric additive was dissolved in a quantity of hydrocarbon base stock to give a definite high-temperature viscosity. In the case of aircraft hydraulic fluids, this viscosity was taken to be 10 centistokes at 130 F. The viscosity of the fluids in centistokes at −40 F was then determined. The blend yielding the lowest viscosity at −40 F was found to contain the polymer of highest molecular weight. This polymer was then considered to have the best blending efficiency. The data given below in

Table 1 illustrate this point for polymers compounded from an 8-carbon atom alcohol.

TABLE 1. Blending efficiency of Acryloid polymers.

Polymer No.	Relative molecular weight*	Acryloid concentration weight per cent	Centistoke viscosity at 130 F	Centistoke viscosity at —40 F
1	94	6.5	10	340
2	84	7.0	10	350
3	49	8.7	10	380
4	43	9.5	10	390

* Defined as the viscosity in centistokes at 210 F of a 30 (weight) per cent solution of the polymer in a standard hydrocarbon base stock.

The above method was found to be very suitable for securing a relative estimate of the blending efficiency of Acryloid polymers. Since low-temperature fluidity is very desirable in hydraulic fluids, it was apparent that a higher molecular weight polymer was the most suitable. This was also desirable from an economic standpoint, since it was apparent that less of the higher molecular weight polymer would be required in the hydraulic fluid.

1.8 LOSS OF VISCOSITY DUE TO SHEAR

The molecular weight of the acrylic ester polymer that could be used was limited, however, by the tendency of the polymer molecules to be sheared and to decrease in apparent molecular weight when subjected to excessive turbulence and throttling under conditions of high-pressure drop. Such conditions prevail to varying degrees in most types of conventional hydraulic equipment employing high-speed pumps, relief valves, etc. The normal result of such a phenomenon was found to be a decrease in the fluid's viscosity, the magnitude of which was dependent on the molecular weight of the Acryloid employed in the fluid. Thus, it became necessary to select an Acryloid having suitable blending efficiency, but whose molecular weight would not cause excessive viscosity decrease due to shear. A testing program was carried out in a hydraulic cycle consisting essentially of a hydraulic pump, loaded by means of a particular throttling or relief valve, through which the oil was cycled approximately 5,000 times at a pressure of 1,200 psi and an oil temperature of 100 F. The data in Table 2 illustrate the effect of Acryloid molecular weight on the permanent viscosity decrease of fluids blended to approximately 10 centistokes at 130 F. The Acryloid polymers were prepared from 8-carbon atom alcohols.

TABLE 2. Viscosity stability under shear.

Polymer No.	Relative molecular weight (as defined above)	Per cent decrease in 100 F centistoke viscosity after 5,000 cycles in pump test
1	46	15
2	50	19
3	63	22
4	77	25

It was decided that 15 to 20 per cent decrease in viscosity would not jeopardize the operation of hydraulic fluids. The tolerable Acryloid molecular weight range, therefore, was controlled by employing only those polymers meeting the requirements of a given mechanical treatment, such as a pump test. The mechanical treatment of hydraulic fluids in various types of hydraulic pumps was undertaken to define more clearly the extent of permanent viscosity decrease due to shear under various test conditions. Table 3 below gives the range of values of permanent viscosity decrease, obtained in different units with a typical specification AN-VV-O-366b (aircraft-type) hydraulic fluid. Each unit was pressure-loaded by means of a balanced-type relief valve.

Because of the increasing tendency towards high-pressure operation (3,000 psi) work was devoted to the study of more stable polymers. In no case, however, did the viscosity decreases listed above hamper the satisfactory operation of the test unit. The mechanical tests proved suitable for judging the shearing action of hydraulic pumps and valves, as well as evaluating the suitability of the Acryloid or polybutene polymers.

During the course of the work, it was found that the polymer-containing hydraulic fluids

[redacted]

would incur a temporary decrease in viscosity when subjected to extremely high-shearing stress in small capillaries or small clearances, usually under the influence of high-pressure drop. This property could be measured quantitatively in small capillaries and was also evidenced by slight increases in internal fluid leakage past close-fitting parts such as pistons and valve plates, caused by this temporary decrease in fluid viscosity. Both polybutene and Acryloid blended fluids displayed this behavior. The magnitude of this temporary viscosity decrease was found to be dependent upon polymer molecular weight and concentration and also fluid viscosity. Molecular weight appeared to be the controlling factor. The effect upon internal leakage was at no time harmful to the satisfactory operation of any hydraulic unit. The control of permanent viscosity breakdown by mechanical means was considered adequate for limiting the extent of the temporary viscosity decrease that prevailed under very high rates of shear, i.e., of the order of 0.5 million reciprocal seconds.

TABLE 3. Viscosity decrease due to shear in pump units.*

Type of Pump	Per cent decrease in viscosity in centistokes at 100 F after 5,000 cycles through unit					
	Hydraulic pump alone		Vickers relief valve		Total for pump and relief valve	
	1,000 psi	3,000 psi	1,000 psi	3,000 psi	1,000 psi	3,000 psi
Pesco gear	10	17	20	27	30	44
Dowty piston	6	15	20	28	26	43
Vickers piston	4	16	23	26	27	42
Hycon piston	3	5	25	35	28	38

* Test conditions: Oil temperatures 100 to 200 F; operating pressure of 1,000 and 3,000 psi and a pump speed of 3,600 rpm.

As the different chemical, physical, and mechanical phases of this project developed, it became evident that it would be possible to produce a polymer-containing fluid suitable for hydraulic systems. An aircraft hydraulic fluid could be made to have a high-temperature viscosity adequate to meet the most severe operating requirements and adverse leakage conditions. The viscosity at −40 F could be as low as 200 to 300 centistokes, which is very much lower than the value of 7,000 centistokes for the 3580-type fluid used in 1941. The new fluid could be prepared with a pour point of −80 F and a flash point above 270 F, and these properties were at least as good or better than those in the previous 3580 fluid. In addition, it was found that many other properties could be incorporated into the basic formula without sacrificing or jeopardizing other important features that were needed. To obtain these properties, however, it was necessary to utilize new types of petroleum base stocks, for they contributed many important properties to the finished fluid. Subsequent work showed that the concentration of the proper type of polymer need only be in the range of 5 to 15 weight per cent. Petroleum stocks could therefore be used to provide the bulk of the needed materials for these improved fluids. Their cost would also be very materially lower.

1.9 BASE STOCKS

A comprehensive study of methods for securing suitable petroleum base stocks was undertaken. It was found that some of the existing manufacturing processes could be simplified, and that some of the anticipated problems could be averted. For example, in order to obtain petroleum fractions free from solids at temperatures of the order of −80 F, it was thought at first that thorough dewaxing would have to be carried out. Such a process would be laborious, expensive, and, above all, it would interfere seriously with the rapid production of these needed stocks because an extensive research and development program would have to be completed to learn how to dewax at these temperatures, since this had never been done before. If this dewaxing had to be done, considerable additional plant facilities and machinery, such as compressors, heat exchangers, filters, and other pieces of mechanical equipment, very much in demand by other war projects, would have been required. These difficulties might have delayed production seriously. However, upon more thorough examination of various crude oils, it was found that there were ample stocks of the so-called wax-free or naphthenic crudes which could be utilized if they could be given some additional refinery treatment. This related to removing the aromatic-type hydrocarbons which are invariably found in such crudes. Aromatic hydrocarbons are particularly deleterious to the synthetic rubbers, which are used for making all sorts of seals in hydraulic equipment. The elimination of these aromatic hydrocarbons at extremely low temperatures proved to be a simpler process than dewaxing. In addition, considerable "know-how" was available on these dearomatization processes, which consisted of either solvent-extracting the desired petroleum fraction, or treating it with sulfuric acid, or both. It was found that the aromatic content of stocks so treated could be controlled by evaluating the aniline point, and the aniline point change, using a standardized laboratory acid-extraction procedure.

Briefly, the preparation of a finished suitable base stock from a typical refinery virgin gas oil or fuel oil requires the use of at least four of the following steps, carried out in approximately the order listed: (1) preliminary chemical treatment to remove deleterious substances such as naphthenic acids, phenols, nitrogen bases, sulfur compounds; (2) distillation to produce the required viscosity and volatility so that evaporation and inflammability requirements could be met; (3) dewaxing if there was insoluble material at low temperatures, but this step, as indicated, is normally not employed; (4) dearomatization to eliminate the aromatic-type hydrocarbon harmful to synthetic rubbers; and (5) clay filtering and blotter pressing to remove any contamination introduced by any of the previous physical or chemical operations.

1.10 DEVELOPMENT OF FINAL FLUIDS

Once the general plan and procedure for making these improved hydraulic fluids was clear, the specific details were worked out methodically with careful attention to the availability of the most suitable and effective ingredients in order to obtain the optimum combination yielding the best overall properties in the finished fluid. An aircraft hydraulic fluid specification was prepared (AN-VV-O-366a, August 6, 1942), providing for the measurement and control of over 20 different properties of the fluid. Commercially prepared fluids meeting this specification were prepared without serious difficulty and delivered promptly.[a]

The improved hydraulic oil for Army Ordnance is described in specification AXS-808 (September 9, 1942). The excellent performance of the aircraft-type fluid in Alaska during the winter of 1942-1943 was reported by the AAF in March of 1943.[8] Army Ordnance carried out extensive firing tests on recoil oil AXS-808 during the winter of 1942-1943 at The Proving Center, Winter Detachment, Camp Shilo, Canada. The results are given in Army Ordnance report No. RLD 6-1. It was recommended that this oil replace those used earlier, and that all future design of recoil mechanisms be based on AXS-808 fluid.

Extensive research and development work was also carried out on an improved Navy ordnance hydraulic fluid. This culminated in the issuance of Navy Specification O.S. 2943 (March 25, 1943). Later (August 1943), a complete revision of another Navy hydraulic oil was made to correct all the deficiencies of this older oil and to bring it up to date with the new developments in hydraulic fluids. This resulted in a completely revised form of Navy Ordnance Specification O.S. 1113 for hydraulic oil.

The development of these three hydraulic fluids led to requests for other low-temperature fluids for use in compasses, and some special compass fluids were developed and produced for the Army Signal Corps. At the same time there were requests for improved damping fluids which were necessary in new fire-control and range-finding equipment being developed by Navy Ordnance. Several damping fluids were prepared which were successful. They solved problems which otherwise would have required much more extensive mechanical and electrical changes in this fire-control and directional equipment. These damping fluids, for the most part, are extremely viscous fluids of a very low volatility with exceptionally flat viscosity-temperature curves. Some of them are prepared by dissolving carefully selected polymers in an ester base stock, such as 2-ethylhexyl sebacate, and incorporating other additives to prevent oxidation, corrosion, etc. Approximately 1,800 gallons of three different types of damping fluid were prepared by the project and consigned to industry as directed by the Navy Bureau of Ordnance. These damping fluids are designated as PRL 1866, PRL-Ac-239, and PRL 1700.[11] In order to get production promptly and to insure the proper quality of these damping fluids, it was found expedient to have them prepared as part of the project.

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[a] In addition to being adopted by the U. S. Army and Navy, this same fluid was adopted by the Canadian Air Force. The Canadian specification is 3GP12 (September 7, 1942). The British Ministry of Aircraft Production also adopted in its entirety the AN-VV-O-366b specification, the British specification being known as DTD-585.

1.11 TACKINESS

In order that adequate quantities of the Army, Navy, and Air Force fluids could be made available at a time when supply was of paramount importance, the specifications were formulated as a sort of compromise between fluid availability and quality. For example, the flash point on the aircraft fluid was placed at 200 F because this value was most consistent with production of base stock in refineries, even though flash points 70 F higher could be produced in laboratory and pilot plant equipment. This increased volatility made it necessary to put additional emphasis on the evaporation characteristics so that when thin films of these hydraulic fluids were evaporated tacky residues would not be formed. When base stocks are too volatile, such tacky films form, because the polymer is being concentrated in them through the loss of base stock by evaporation. To overcome this, it was necessary to have present in the finished fluid a certain amount of a relatively nonvolatile oil so that the evaporated films were oily and not tacky. This was accomplished by having 25 per cent, or more, of a relatively high-boiling and nonvolatile oil present in the finished fluid. This expedient eliminated most of the difficulties that might otherwise have been encountered. Tacky or sticky oil films might have caused delicate mechanisms, such as valves and hydraulic controls, to become sluggish or fail to move, since many of these operate with very small forces.

1.12 GASKETS

The problem originally assigned by the Air Forces included the development of gasket materials. Because of the impracticability of replacing the gaskets in the hydraulic systems of many existing airplanes, it was considered more expedient to require that the new fluid be operable with the gaskets then in use. For this reason the specifications defined the minimum swelling and shrinkage that would be tolerated by all types of rubber sealing media. As an additional control, the aniline point and aniline point change were incorporated into the specification to limit the concentration of aromatic hydrocarbons that would be tolerated in the finished fluids, since it was found that these hydrocarbons were much more harmful to the rubbers than the paraffinic or naphthenic types. Experience showed that fluids compatible with the rubbers then in use could be produced satisfactorily. Accordingly, this type of control was extended to the Army Ordnance and Navy Ordnance hydraulic fluids.

1.13 SUMMARY OF SPECIFICATIONS

Table 4 summarizes the specifications of the new hydraulic and recoil oils. It also compares the properties of the new aircraft oil (AN-VV-O-366b) with that of the previous aircraft hydraulic oil known as specification 3580. It should be noted that the latter two oils have the same viscosity at 130 F. Above this temperature the AN-VV-O-366b oil is more viscous. This is desired in order to improve lubrication, reduce leakage, and permit hydraulic pumps to operate at pressures up to 3,000 psi. Below 130 F the new aircraft oil becomes less viscous as the temperature is decreased. This is also desired because it improves the low-temperature performance of equipment operated by this oil. The reduction in low-temperature viscosity is evidenced by comparing the two viscosities at −40 F. In the case of the 3580 oil, the value is around 7,000 centistokes, whereas for the AN-VV-O-366b oil the specification requires that the viscosity be less than 500 centistokes. The effect of this decreased viscosity on performance will be outlined subsequently. The ASTM slope listed is the actual negative slope on the ASTM special viscosity graph paper designated as D341-39 of a straight line drawn between the points on the actual viscosity curve at −40 F and at 130 F. The viscosity index is also tabulated for comparative purposes, but it has doubtful meaning in this higher range. As already mentioned, specification AN-VV-O-366b represents a compromise between the best fluid which can be made and one which is relatively easy to prepare and produce in quantity. With existing refinery equipment to produce the base stocks and with the available Acryloid types, it is possible to produce a similar fluid with a maximum of 500 centistokes at −60 F in place of −40 F.

Table 4. Summary of hydraulic fluid specifications.

Property	Aircraft hydraulic oil 3580-C	Aircraft hydraulic oil AN-VV-O-366b	Navy hydraulic gear oil O.S. 2943	Army recoil oil AXS-808 Rev. 1
Min. Centistokes at +210 F	(4.2)	(5)	10	15
Min. Centistokes at 130 F	(10)	(10)	(19)	(35)
Min. Centistokes at +100 F	18.5	(14)	27	55
Max. Centistokes at 0 F	(500)	(100)	215	(730–820)
Max. Centistokes at −25 F	(2200)	(260)	600	(2600–3200)
Centistokes at −30 F	(3000)	(320)	(750)	(3400–4400)
Max. Centistokes at −40 F	(7000)	500	(1200)	(6700–9500)
ASTM slope	(0.73)	0.56	0.45	0.49
Approx. viscosity index	155	225	190	165
Min. flash point (C.O.C.), F	280	200	225	210
Min. fire point (C.O.C.), F	....	(210)	235	220
Max. cloud point, F	....	−65	−35	−40
Max. pour point, F	−50	−75	−40	−50
Max. shear breakdown, per cent	....	15	25	25*

* As compared to 25 in reference oil. Values in parentheses are approximate.

1.14 BLENDING PROCEDURE

The procedure in blending the petroleum base stock with Acryloid to produce an oil meeting the viscosity-temperature requirements of AN-VV-O-366b is illustrated in Figure 1. The viscosity at 130 F is plotted versus the ASTM slope on semi-logarithmic graph paper. Point *C* represents the blend of the light base stock *B*

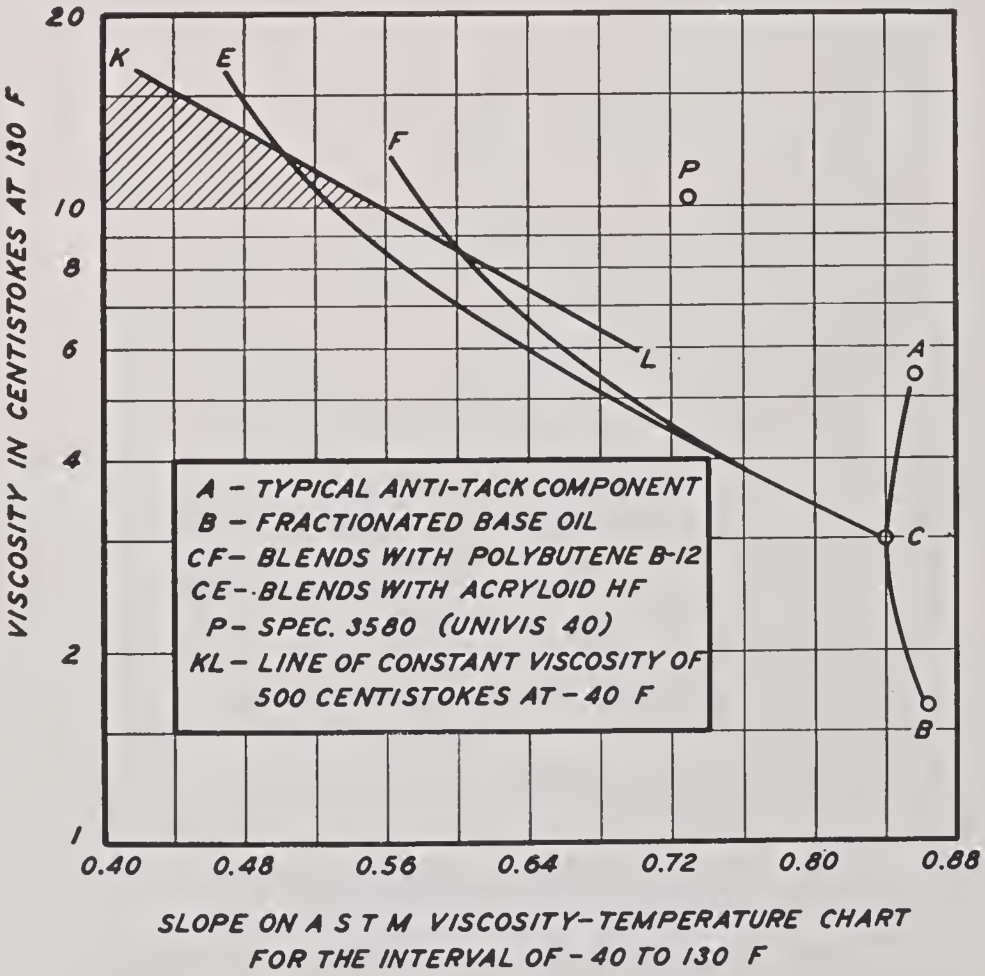

Figure 1. Effect of polymer addition in the blending of hydraulic fluids.

with the anti-tack component *A* having a flash point of 300 F or higher. Addition of polymer causes the point representing the mixture to move upward and to the left along lines *CE* or *CF*, depending on whether Acryloid or polybutene is used as the polymeric additive. AN-VV-O-366b hydraulic fluids must have a viscosity at 130 F of more than 10 centistokes, and at −40 F of less than 500 centistokes. Line *KL* represents the latter requirement. It is evident that a fluid meeting the viscosity requirement of AN-VV-O-366b oil must lie within the shaded area of this figure. In other words, starting, for example, at point *C*, the ASTM slope is decreased by the addition of polymer along the line *CE*, or along line *CF*. For the particular base stock illustrated, and designated as *C*, it can be seen that the viscosity-temperature requirements can be attained in this case using the Acryloid polymer but not with polybutene. It is also evident that if the viscosity of the base stock were somewhat lower, polybutene could provide the required viscosity-temperature characteristics. Point *P* typifies a fluid of the 3580 type.

1.15 SERVICE PERFORMANCE

One important criterion for judging the value of these new hydraulic fluids is obtained by analyzing their service behavior. The National Research Council of Canada obtained comparative data on the rotation of a Bristol gun turret and a Frazier-Nash turret, and on the depression of a Bristol gun and that of a Frazier-Nash gun.[7] These data were obtained in a low-temperature altitude chamber. Records were made of rotation and depression times when employing first a fluid designated as DTD-44-D and secondly AN-VV-O-366a fluid. The former is similar to the old 3580 fluid and matches the viscosity of the AN-VV-O-366a fluid at about 130 F. Figure 2 typifies the data obtained on the rotation of the Bristol gun. With the older type of fluid, the gun was essentially inoperable at −35 F, and the rotation time was excessive when starting from the cold at −20 F. The use of the AN-VV-O-366a fluid made the gun operable at any temperature normally encountered, even at high altitudes. Although the new fluid cannot be claimed to be phenomenally better than the old one, the improvement is sufficient to make the difference between the gun's working and not working.

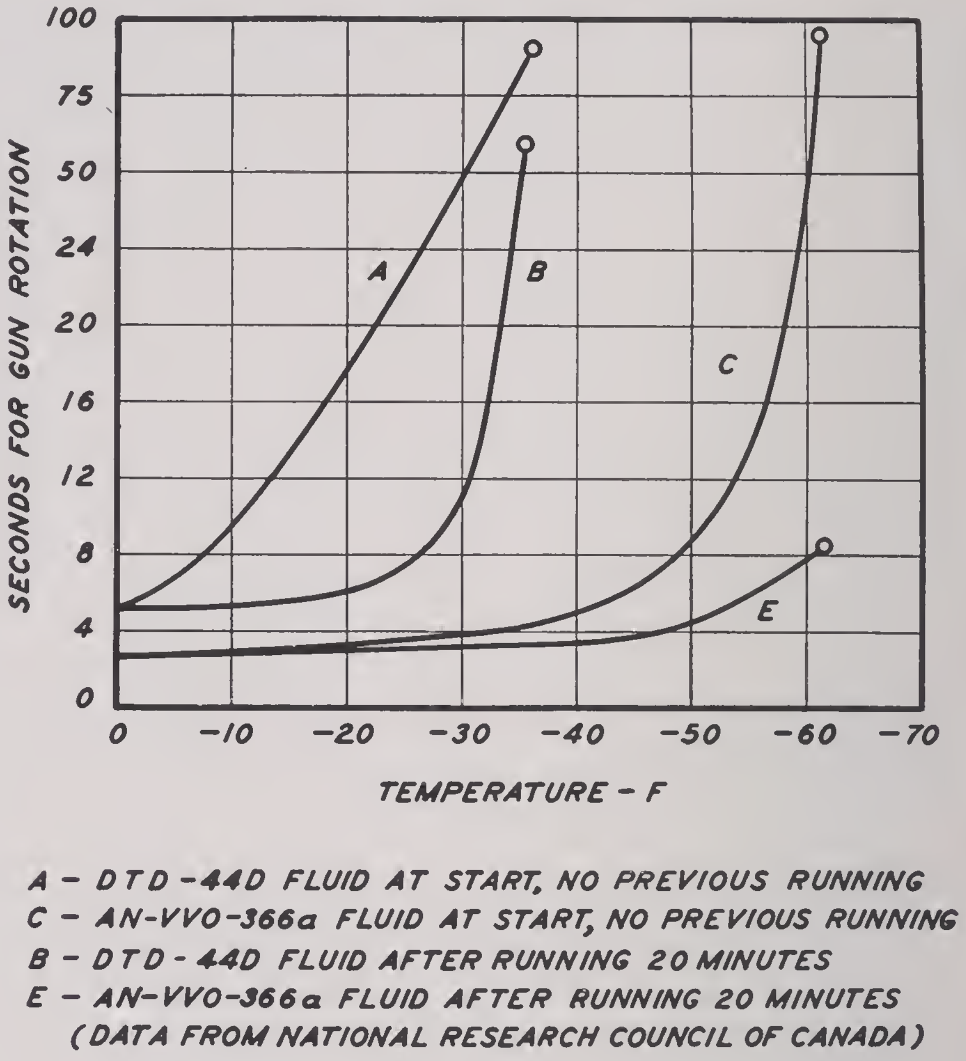

FIGURE 2. Effect of improved hydraulic fluids on the time of rotation of a Bristol gun.

Tests with other aircraft equipment at low temperatures showed similar results. Cold-room tests with Sperry upper local turret using a Vickers drive unit and a standard shunt motor showed that the minimum starting temperature with 3580 oil was 0 F. Using AN-VV-O-366a fluid, starting was possible at −70 F. The same unit with a pre-excited field shunt motor started at 0 F or above with the 3580 oil and at −70 F or above with the AN-VV-O-366a fluid. Using a compound wound motor on the same installation, the torque was so increased that the starting temperatures were lowered to −55 and −100 F, respectively. The AAF reported[8] the excellent results that were obtained with the new fluid in Alaska during the winter of 1942-1943. In addition to these low-temperature tests, samples of used oils from different aircraft have been obtained and analyzed carefully. In each case it was found that there was very little change occurring in the fluid. There was every reason to believe that it would have a very long service life. Such data are important in determining whether the viscosity-shear properties, oxidation stability, evaporation characteristics, etc., have been adequately provided for in these specifications.

The recoil oil AXS-808[11] was tested in 37-, 75-, and 155-mm guns, 75- and 155-mm howitzers, and 3-in. and 90-mm antiaircraft guns. In nearly 100,000 rounds of proof firing its performance was satisfactory throughout a temperature range of −40 to +150 F. This oil made it possible to consolidate several Army Ordnance specifications for recoil and hydraulic oils.

Extensive laboratory and service tests were also conducted on the new Navy Ordnance hydraulic oil O.S. 2943 and the revised form of O.S. 1113 hydraulic oil. These tests showed that the former oil would permit operation of hydraulic drives on guns at temperatures that were from 30 to 40 F lower than with the O.S. 1113 oil. At elevated temperatures, the O.S. 2943 oil performed at least as satisfactorily as the O.S. 1113 oil. The Navy employed both hydraulic oils because at the start of the work it was not possible to get the required quantity of the O.S. 2943 oil. The new Navy hydraulic oils produced better operation of the hydraulic equipment, retarded or prevented rusting, and extended materially the time between shutdown or cleaning periods. These Navy hydraulic oils contained about 1 per cent of tricresyl phosphate. This was used as an anti-seize additive if close-fitting mechanical parts tend to weld together under severe usage.

1.16 NONINFLAMMABLE FLUIDS

Initially the main emphasis was on the development of hydrocarbon base fluids. There were two reasons for this. First, there was a critical demand for an improved fluid. The early work showed considerable promise; it indicated that this approach was the one most apt to succeed in the shortest possible time. Second, the Air Forces stated that they wished to retain hydrocarbon base fluids which would not only be miscible with the previous fluids then widely used, but which would also be operable in contact with the gasket materials and other sealing devices installed in the many thousand airplanes in the field. In other words, it was imperative that the problems relating to the deficiencies in the hydraulic fluids used at that time be solved promptly, with no change in the actual hydraulic systems.

The principal, if not the only disadvantage of a hydrocarbon base fluid is its inflammability. This property has caused considerable concern at various times and there have been several requests from the Services for a noninflammable fluid. It proved particularly difficult to obtain objective data on the real importance of having a noninflammable fluid. It is not yet known whether the substitution of such a fluid for the present oil would reduce significantly fire hazards in airplanes and other fire losses. Laboratory tests of inflammability appeared to be of little value since carbon tetrachloride can be made to burn under favorable conditions, whereas an incendiary bullet passing through a metal container of the present hydrocarbon base hydraulic fluid will not cause a fire if the container is not under pressure and if the incendiary passes below the liquid level. Fragmentary data from the field indicated that aircraft fires resulted in losses which would not have occurred if the fluid had been completely noninflammable. An informal report from an operations analysis group studying bomber losses by the Eighth Air Force indicated that about 4 per cent of the losses incurred during the last half of 1944 would have been avoided if the hydraulic fluid had been noninflammable. Although this percentage is not high, it indicated that, if the figures were reliable, a noninflammable fluid would have saved 5 to 10 bombers and their crews per month during the last half of 1944. Because of these reports the experimental work included a considerable effort directed toward the development of a noninflammable fluid and one that could be made available promptly. Although the results have shown promise, it has not yet been possible to produce a fluid of this type fulfilling all of the various requirements which must be met and which, with the exception of the inflammability, are contained in the present specifications for hydrocarbon base fluids.

The newer silicone oils are much less inflammable than hydrocarbon oils. However, they lack good lubricating properties for steel on steel under adverse operating conditions; they tend to creep more on metals and so there is usually more leakage in the hydraulic system; they shrink rather than swell the present rubbers used in hydraulic equipment (this is objectionable), and their oxidation products are much more corrosive than those from mineral oils. Their production was very limited. The chemistry involved in their manufacture is so complex that it would have been very difficult to obtain adequate supplies promptly.

Ethylene glycol-water mixtures with various additives showed promise, as did β-(methoxy-methoxy) ethanol and certain polyglycol-mixed ethers produced by the Carbide and Carbon Chemicals Company. Although all these are inflammable in the strict sense of the word, they are considerably less inflammable, particularly when water is present as a component of the fluid, than the hydrocarbon base fluids. Triethyl phosphate mixtures also have relatively low inflammability, but this ester is readily hydrolyzed by water to produce very corrosive products. A research program carried out by the Standard Oil Development Company with the support of the Bureau of Ships led to the development of various types of halogenated hydrocarbons showing some promise, but none met the specifications of the present hydraulic fluids; even these could be made to burn under certain conditions. An important objection is their toxicity. This would definitely make them undesirable for use in closed and confined spaces where hydraulic equipment is frequently installed. The carbon-fluorine compounds under development for various war uses may later become available in sufficient quantity to warrant consideration as fluids for hydraulic mechanisms. Their properties were not studied in detail in this connection largely because they were not available in the quantities needed.

Of the various non-hydrocarbon types studied, the esters of certain dibasic acids having molecular weights of about 300 and higher, showed good viscosity-temperature properties and other characteristics useful in hydraulic fluids. A typical example is 2-ethylhexyl sebacate. This ester has a viscosity of about 12 centistokes at 100 F, a freezing point of −55 F, a flash point of about 440 F, and a viscosity index of about 150. Esters of this type may be employed with certain Acryloids to obtain viscosity indices of 180 to 200, and these materials appear to be good lubricants from many standpoints. Lubricants employing this ester together with certain additives are being developed as a special instrument oil. Many other laboratories are using these esters as the basis for making various special low-temperature greases having exceptional fluidity at low temperatures, but without the attendant evaporation difficulties now present in greases derived from conventional mineral oils. This project was the first to point out the valuable properties of this class of esters.

Various water-soluble organic solvents have shown promise, especially when mixed with special polymers to improve the viscosity index, or to reduce the ASTM slope, and to attain the viscosity properties which are needed. Solvents tested included ethylene glycol, propylene glycol, triethylene glycol, dimethoxy tetraethylene glycol, β-(methoxy-methoxy) ethanol, and methyl carbitol. Thickeners included starch acetates, polyvinyl alcohols, carbowax, sodium carboxymethyl cellulose, and certain Acrysols made by Rohm and Haas. A typical water-base fluid, approximating the AN-VV-O-366b specifications, contained 33 per cent water, 51 per cent β-(methoxy-methoxy) ethanol, 9 per cent triethylene glycol (as anti-tack agent), and 7 per cent Acrysol. Such a fluid had a viscosity of 10 centistokes at +130 F and about 1,200 centistokes at −40 F. The freezing point was −65 F.

1.17 FUTURE RESEARCH

The field of non-hydrocarbon organic and water-base fluids has not yet been explored adequately, and it is probable that continued effort would lead to the development of fluids which would have all desirable properties of hydrocarbon base fluids and yet have the additional advantage of being noninflammable. The record of the development of the hydrocarbon base fluids has emphasized, however, that the selection of a fluid with a satisfactory viscosity-temperature property is only a starting point. It may develop that the corrosion, evaporation, toxicity, lubrication, and other specific properties will be much more difficult to attain than in the case of the hydrocarbon base fluids. Work on developing higher flash or noninflammable fluids meeting the present specifications should continue, in addition to that which is directed toward the perfection of the present fluids with respect to improvements in antirust properties, oxidation and shear stability, rubber swelling, tackiness and evaporation, foaming, leakage, lubrication, etc. The development of improved gasket materials should also parallel the work on the fluids, for improvements in some of the new high-polymer plastics may enable gasket materials to be manufactured which are insensitive to any of the fluids which may be subsequently employed.

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Chapter 2

DUST REMOVAL FROM AIRCRAFT ENGINE AIR SUPPLY

Operations in North Africa in the latter part of 1941 emphasized the necessity of removing dust and sand from the air intake of military aircraft engines. Under desert conditions aircraft were found to have a service life as short as 5 to 15 hours. Efficient fabric and metal cloth filters had been developed, but these required servicing or replacement, and introduced a serious pressure drop in the air-intake duct. Such filters were applicable in air ducts of a large cross section, but many of the aircraft had very small air ducts in which filter installation was impractical. Many aircraft had been sent to Africa without filters, and it was desired that an air-cleaning device be developed which might possibly be installed in the field. The problem was brought to the NDRC by the NACA in late March 1942.

A preliminary analysis of the problem and discussions with the Bureau of Aeronautics and Naval Aircraft Factory indicated that filters for the purpose were in a relatively advanced state of development, and that much more could be done by the Services in adapting existing filters for combat aircraft than by the NDRC in developing improved filter designs. Accordingly, work was initiated on the development of a simple device employing the principle of centrifugal separation, with the idea that it might either replace or complement the filter.

Small-scale experimental work was carried out to determine the possibilities of a duct skimmer, a cowling skimmer, and a channel air filter. Half-scale tests were carried out on the cowling skimmer, which proved to be the most promising of the three small devices. The duct skimmer consists of a splitting vane positioned in the duct following a bend, with provision to withdraw and reject the dust-laden air collected along the outer curved wall. This device was tested quite thoroughly on a small scale, and the final report contains detailed data on its performance. It was finally rejected for several reasons, including the following. On idling the duct may be under suction, so that dust-laden air would not be rejected; the air velocity in the duct is too low to make the device operate efficiently when the engine is idling; it is not applicable to all aircraft duct designs; and it is less efficient than the simpler cowling skimmer.

The channel air filter consisted of a cloth filter fitted diagonally lengthwise in the air duct, with the idea that the high-air velocity parallel to the filter surface would make the filter surface self-cleaning. A part of the air was withdrawn and rejected at the end of the cloth on the upstream side in order to maintain the desired high velocity over the dirty surface. It was found, however, that dust continued to build up on the cloth surface, and that relatively little was swept off. This led to excessive pressure drops with reasonable filter areas, and the device was abandoned.

The cowling skimmer consisted of a vane on the cowling, inclined toward the rear of the plane and blocking the normal air passage directly back through the air duct (see Figure 1). The propeller wash tended to sweep the dust past the vane and allow clean air to be drawn into the duct through a slot behind the up-tilted vane. The principle of this device was first tested by employing a tee skimmer in which cleaned air was withdrawn through a right-angle connection to a horizontal straight-through passage carrying dust-laden air. This small device was later modified to incorporate a tilted vane partially covering the outlet to the

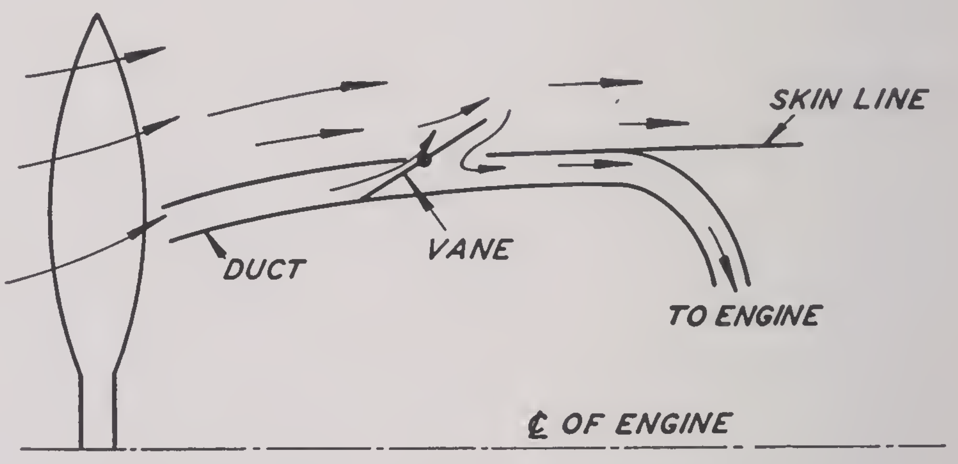

Figure 1. Cowling skimmer for removal of dust from aircraft engine air intake.

tee, and later a half-scale model was constructed with a vane 5 in. wide and 3½ in. from pivot to trailing edge, with the top edge of the vane 1½ in. above the skin. Approach velocities varied from 48 to 102 ft per sec and velocities in the vane opening were varied from 21 to 102 ft per sec. The data were correlated in such a way that only a small extrapolation was necessary to obtain the estimate of performance on a full-scale installation shown below.

Condition of Operation	Estimated Cleanup Efficiency, in Per Cent, for Particles of Micron Size				
	10	20	43 (325 mesh)	74 (200 mesh)	417 (35 mesh)
Take-off	55	71	88	96	99
Taxiing	65	78	93	98	99
Idling	70	81	94	99	99

The principal characteristics of the device are:

1. Efficiency is best under conditions of low-engine load, when the ratio of intake-duct speed to propeller wash speed is low.
2. Efficiency increases rapidly with increase in particle size, the device being highly efficient for coarse dusts.
3. Pressure drop is low.
4. The vane can be readily displaced to obtain normal intake with ram effect.
5. The performance does not vary with use.
6. No cleaning or servicing is required.
7. It is conceivable that the device could be installed in certain planes in the field.
8. If used with a filter, it removes the coarse dust which the filter has difficulty in retaining, and greatly reduces the required frequency of cleaning of the filter.

The results of the experimental work on the cowling skimmer were brought to the attention of the NACA, the Bureau of Aeronautics, and Army Air Forces in late 1942, and the final report issued February 4, 1943.[1] By this time standard requirements for filter installations had been set up, but it was found possible to employ the cowling skimmer principle in various aircraft installations. The skimmer is more effective than the filter for the larger particle sizes; therefore the two are frequently employed to complement each other, with air intake to the filter designed to bring in air in a direction normal to the line of flight, and dampers installed to by-pass the filter and obtain the ram effect on air intake after take-off. The NDRC report was made available to aircraft manufacturers in order that they might see how best to employ the skimmer principle in conjunction with a filter installation, thus greatly prolonging the time interval between required cleanings of the filter.

Chapter 3

PROTECTION OF AIRCRAFT FUEL TANKS AGAINST EXPLOSION

3.1 SUMMARY

THE GASOLINE VAPOR-AIR mixture present in partially empty aircraft gasoline tanks represents a serious explosion hazard at moderately low temperatures. To provide an inert atmosphere, a system employing engine exhaust gas was installed in a C-46 airplane and was proved by flight tests involving rapid dives from high altitudes. The complete installation for a twin-engine aircraft such as the C-46 with a fuel capacity of 1,440 gallons has a total weight of approximately 25 pounds, and provides continuous protection.

3.2 EXPERIMENTAL WORK

Mixtures of gasoline and air are explosive when the volume per cent gasoline vapor lies in the range of about 1.5 to 6.0. Since the air in the vapor space over the fuel tends to become saturated with gasoline at the tank temperature, there is a range of operating temperatures over which the vapor space over the fuel will contain an explosive mixture. With aviation gasoline this temperature range is roughly −9 to −29 C at atmospheric pressure and −30 to −50 C at 30,000 ft. Partially empty fuel tanks in these temperature ranges represent serious explosion hazards, as they carry large volumes of explosive gases.

Experimental tests with both incendiary and ball ammunition show that the explosion of a fuel tank containing an explosive fuel-air mixture is quite violent, and that the force of the explosion is doubtless sufficient to wreck an aircraft in flight. Although the Services have received relatively few reports on gasoline tank explosions, it has been supposed that an unknown number of aircraft may have been lost as a result of such explosions, but the reason for the loss was not determined. The probability that important losses resulted from fuel tank explosions seemed sufficient to justify experimental work on the development of means for protecting against the occurrence of explosive gas mixtures in the gas space over the fuel.

Exploratory work was carried out on the generation of inert gas by complete combustion of carbon to form nitrogen and carbon dioxide. A second proposal studied was the removal of oxygen from air by reaction with hot copper, and regeneration of the copper by subsequent reduction of the copper oxide by reaction with gasoline. In each instance it was planned that the oxygen-free gas would be fed into the fuel tanks to replace the gasoline withdrawn to the aircraft engines.

The carbon burner was designed to use specially activated carbon supplied by the Dow Chemical Company. This had the advantages of a low ignition temperature (200 C) and a low ash (1.5 to 3.5 per cent). It was estimated that the weight for carbon and generator would be about 90 pounds for a two-engine ship in normal flight. Greater weight would be necessary to give protection during dives. This unit was considered too hazardous for use because of the temperatures reached, which were well above the melting point of aluminum. If hit by a projectile, the results would be as serious as a hit on an unprotected fuel tank. No satisfactory method was developed for starting or stopping the operation.

The copper regenerative unit depended on the removal of oxygen from the air by copper gauze previously activated with ferric nitrate. This reaction occurred at a temperature of 600 to 700 C. The copper oxide formed was then reduced to the metal with gasoline vapor, requiring about 1 gallon of gasoline for 3,000 to 4,000 cu ft of inert gas produced. This unit would be easy to start and stop, and control would be simple. However, the weight was considered too great. It was estimated that 50 pounds would be required to protect a two-engine plane (2,000-hp engines; 1,440-gallon fuel capacity) during level flight, and this would have to be increased for protection during dives.

The disadvantages of the two systems made them appear less promising than the use of inert gas obtained from the engine. Accordingly, the further work and flight testing were confined to development of the use of exhaust gas. Similar work was carried out simultaneously at Farnborough in England, and the Russians were reported to have had a system using exhaust gases in operation on their Lagg-3 aircraft in 1943.

Exhaust gases low in oxygen may be employed in two ways to provide protection for fuel tanks. The gases may be forced through the gas space over the fuel to purge the tanks continuously, or the gases may be drawn into the tanks only as needed to replace the fuel used, or to maintain ambient pressure in the tank during descent. In either case the hot engine exhaust gases must be cooled and corrosive constituents removed. The experimental work involved ground and flight testing of both systems, referred to as open and closed.

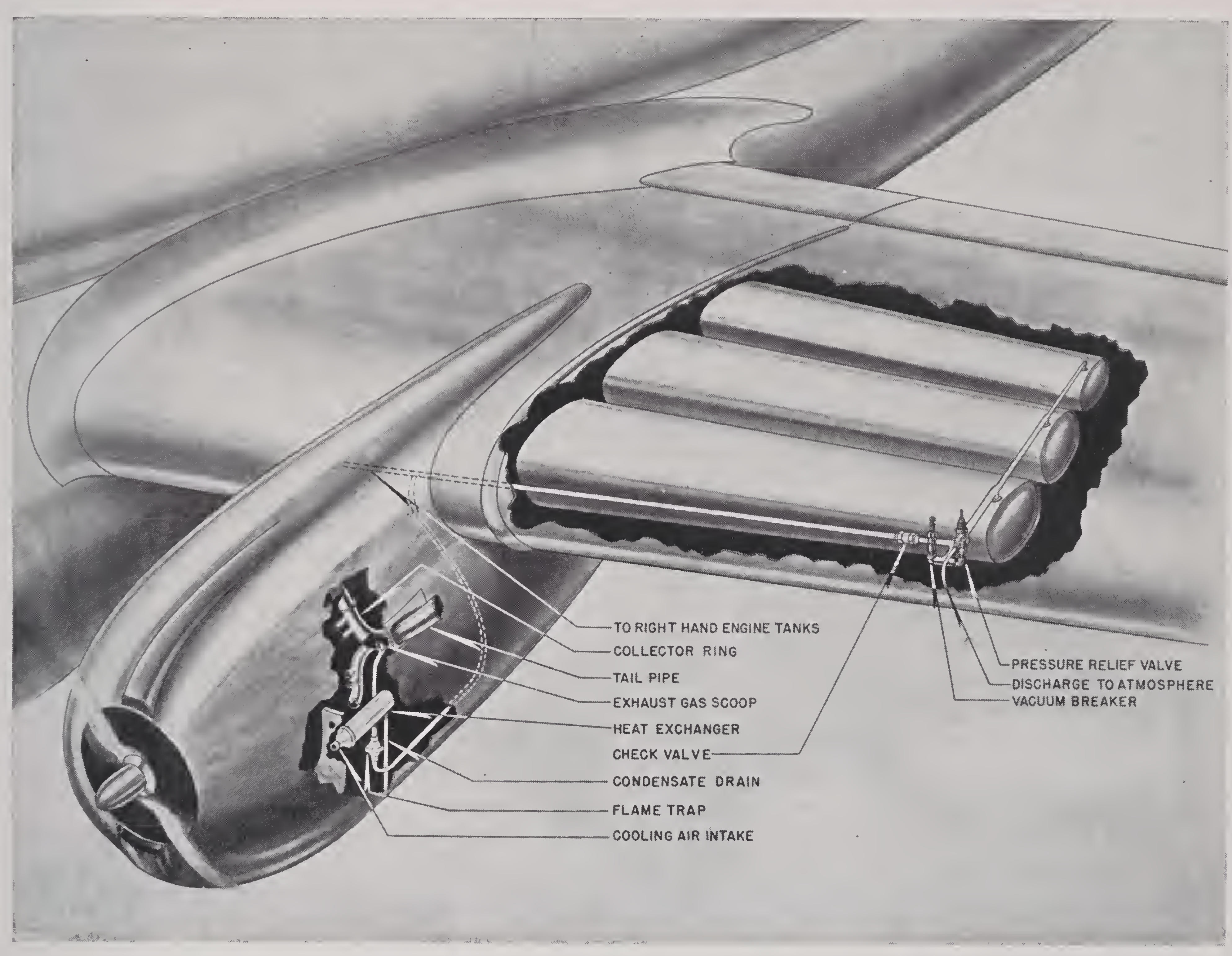
TO RIGHT HAND ENGINE TANKS
COLLECTOR RING
TAIL PIPE
EXHAUST GAS SCOOP
HEAT EXCHANGER
CHECK VALVE
CONDENSATE DRAIN
FLAME TRAP
COOLING AIR INTAKE
PRESSURE RELIEF VALVE
DISCHARGE TO ATMOSPHERE
VACUUM BREAKER

FIGURE 1. General arrangement of installation for using exhaust gases to provide protection for fuel tanks.

The system developed included an exhaust gas scoop placed inside the engine exhaust pipe, a heat exchanger, a flame arrester, gas lines to the wings, a check valve, a vacuum breaker, and a pressure relief valve. The general arrangement of these in the installation as proposed for a C-46 (Navy R-5-C) is shown in Figure 1. For experimental purposes, such a system was set up and tested on the ground, using C-46 wing tanks and exhaust gas and cooling by propeller wash from an aircraft on the ground. The equipment was then placed inside the fuselage of a C-46 and flight tested in dives from 25,000 ft. Two half-tanks were placed one above the other with provision to drain fuel from one to the other at various rates simulating fuel consumption by engines. Rotameters were employed to measure inert gas flow rate, and gas samples were withdrawn from both ends of the upper test tank. Various temperatures were recorded by means of thermocouples and careful measurements were made of the pressure differential between inside and outside of the test fuel tank. Flight test data were obtained with both the closed system, fitted with a vacuum break and pressure relief valves, and the open system, in which these valves were omitted.

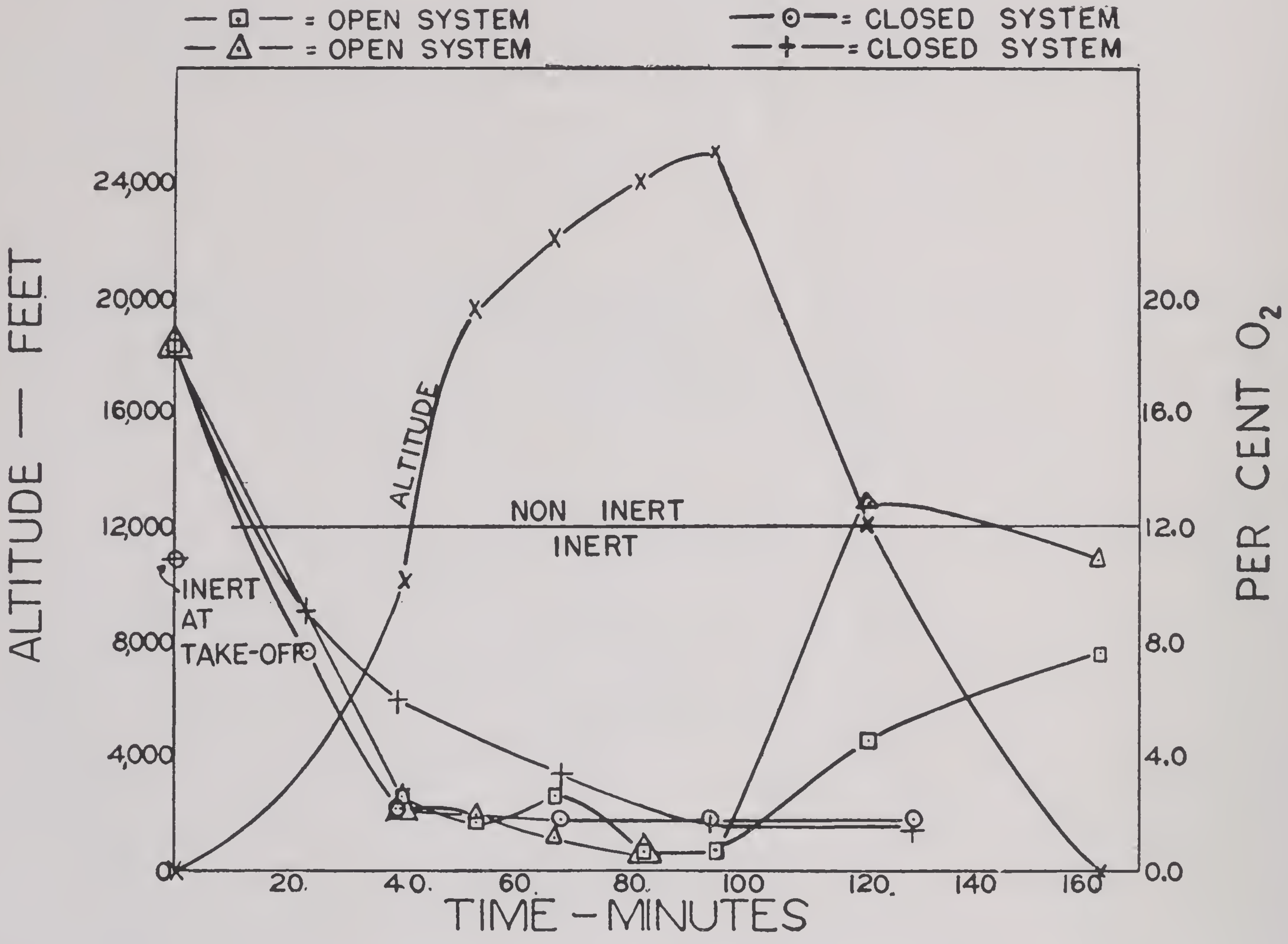

FIGURE 2. Contrast of open system versus closed system.

Four heat exchangers were tested and evaluated. The best performance was obtained from a cylindrical type with straight-through passages for the cooling air. The hot gases were fed through the annular space between two concentric cylinders forming a hollow shell. This shell was supported within a cylindrical air passage in such a way that cooling air passed over both inner and outer surfaces of the hollow cylindrical shell carrying hot gas. This exchanger was constructed of 18-8 stainless, and was so designed as to be easily taken apart for cleaning. The amount of exhaust gas cooled to approximately ambient air temperature was roughly 3 per cent of the exhaust from one engine, and the pressure drop due to friction on the exhaust gas side of the heat exchanger was less than ¼ in. of water.

Figure 2 shows typical results of flight testing with both open and closed systems. The horizontal line at 12 per cent oxygen indicates the approximate composition of the lower explosive limit of air-vapor mixtures, for comparison with the gas compositions obtained in flight by analysis of the gas withdrawn from the fuel tanks. With the open system this limit was exceeded following a dive, because of the fact that the inert gas supply was inadequate to purge the tank under these conditions and air entered through the vents. With the closed system the maximum negative pressure reached during a dive was ¼ in. of water, noted in one dive from 25,000 to 13,000 ft at a rate of descent averaging 2,000 ft per minute.

The flame arrester was a conventional type consisting of alternate flat and corrugated flat sheets of cupro-nickel alloy forming a honeycomb grid. The check valve in the gas line was a ¾-in. fuel line check valve as used on the P-40 aircraft and served to prevent the return of fuel or gas from the fuel tanks. This valve operated under a pressure differential of less than ¼ in. of water. The fuel tanks were standard C-46 all-riveted wing tanks cut in half, and contained the usual baffles, sumps, and bulkheads. The vacuum break was a check valve similar to that employed in the gas line to the tank, but mounted in a vertical position. The pressure relief valve was a modified commercial high-pressure relief valve. The complete installation for a twin-engine aircraft such as the C-46 with a fuel capacity of 1,440 gallons is estimated to have a total weight of 25 pounds.

Considerable attention was given to the possibility of corrosion of various parts of the system by the corrosive condensate or by the hot gases. It was found that corrosion difficulties could be avoided if the scoop and heat exchanger were made of 18-8 stainless. The fuel tanks were cut open after the test program was completed and no evidence of corrosion found. British tests have confirmed these observations.

The possibility of vapor lock due to carbon dioxide dissolved in the fuel has been considered, but British tests with fuel saturated with inert exhaust gas at atmospheric pressure show rather conclusively that vapor-lock difficulties are not aggravated.

The ground and flight tests proved the inert exhaust gas system operable and provided sufficient technical background and experience to serve as a basis for the design of similar systems for installation on combat aircraft. The older systems, employing liquid carbon dioxide in pressure bottles, provided protection for only a few minutes of combat, yet weighed more than the exhaust gas system providing continuous protection and requiring less servicing. The data seem entirely convincing in showing the superiority of the exhaust system over that using liquid carbon dioxide.

Whether the exhaust gas system be of the closed or open type is apparently a question of secondary importance. The open system is simpler because it requires no vacuum break or pressure relief valve, but the gas cooling capacity of the exchanger must be greater. The open system requires purging before take-off with a half-filled tank, whereas the closed system maintains an inert gas over the fuel when the aircraft is on the ground with the engines not running. Failure of the vacuum break on the closed system might cause the collapse of the lining of a safety fuel tank. The various advantages and disadvantages of the two systems will be clarified only by continued flight test of installations of both types.

It seems possible that a small electrically heated catalyst might be developed for installation in each tank, serving to burn out the oxygen of the entering air by nonexplosive reaction with gasoline vapors. No progress was made in following up this idea, but it may well form the basis of future research.

Chapter 4

PYROTECHNICS—FLARES; PHOTOFLASH BOMBS

4.1 SUMMARY

A STUDY OF VARIATIONS in the chemical composition of flares showed the candlepower-seconds obtained per gram of metal-oxidant mixture to be subject to little improvement. For antisubmarine use, a satisfactory underwater flare was developed, burning at the rate of 10 sec per in. with a candlepower of 60,000 in water for the 4-in. diameter size. Colored flares were developed for use at high altitudes in tracking guided missiles, and special igniters produced for this and related purposes.

Various photometric, spectrographic, and high-speed photographic techniques were developed for the testing of photoflash bombs, and the instruments constructed were employed in connection with AAF-Army Ordnance development of better bombs. Bomb-casing strength was shown to be an important factor, and optimum designs were developed on the basis of the test data. The British Mk III aluminum dust bomb was tested and the principle adapted for use in improved bombs of United States design.

Exploratory tests of shock-wave flashes show promise justifying further study.

4.2 FLARES

4.2.1 Introduction

During World War II various new uses for flares were envisaged; for instance, as a means of underwater illumination and for certain high-altitude operations from aircraft. The several types of flare requested by the Services were sufficiently varied to require a rather broad investigation of the field, and this resulted in the development of a number of principles of pyrotechnic behavior which are perhaps applicable beyond the original boundaries of the investigation.

A very difficult matter to improve was the light-producing efficiency of the Service stars and flares, although significant improvements in other characteristics were made with relative ease. Star shells and flares are normally used for visual observation, and hence should be judged by their effect on the human eye. Therefore, all light measurements were made with a barrier-type photocell filtered to give a spectral sensitivity close to that of the human eye, so that the light units were actually eye units. The reference standard of illumination was a tungsten filament lamp, operating in the neighborhood of 3000 K and generating some 4,000 candlepower.

It was demonstrated that the efficiencies (candlepower-seconds per gram) are very nearly constant for a series of flares having essentially the same spectral distribution and percentage metal content, but that candlepower and burning speed could be made to vary widely in an inverse relation to each other by changes in composition of the flare mixture.

4.2.2 The Underwater Flare

At the time of the greatest activity of German submarines along the eastern coast of the United States, the Navy requested flares which would burn under water and illuminate or silhouette a submerged submarine at night. Detection devices used on blimps gave the approximate position of the submarine, but it was believed that visual aid would make bombing much more accurate. Before the development was completed, the German submarines had moved away from the coast into deep water and the development was stopped. The chemical work was finished, however, and preliminary tests made on the mechanical design.

It was obvious that a flare to burn under water must carry enough oxidant in its mixture to burn all the metal present, if the maximum efficiency of operation was to be attained. All mixtures studied for use in underwater flares employed stoichiometric mixtures of metal and oxidant, in recognition of this principle.

Since all light-producing chemical reactions of an underwater flare would occur within the sphere of combustion that could be maintained immediately over the burning flare surface, and since the intensity of illumination would depend primarily upon the size of that sphere, an effort was made to insure complete combustion of the metal as near the flare surface as possible. This was accomplished by using finely divided ingredients, 200 mesh, or finer. There are several other advantages to the use of finely divided flare ingredients: the combustion is smoother because a more homogeneous mixture is obtainable; the possibility of flare failure from water seepage into the cake is minimized, since the voids to be taken up by the binder are small; the resistance of the cake to mechanical damage is considerably increased.

Extended tests of the Navy's standard star mixture[a] were carried out to determine its usefulness as an underwater flare, and it was concluded that the mixture was not suitable for such a purpose.[5] The Navy mixture characteristically began to burn with an inferior illumination, and developed into a remarkably brilliant flare after some 15 sec. Tests of the mixture and variations of the formula indicated that the primary difficulty may have been the paraffin binder used. It was shown that boiled linseed oil with a drying catalyst of powdered pyrolusite was generally superior to paraffin, shellac, or nitrocellulose as a binder. One possible binder of considerable appeal was suggested but was never tested; namely, furfural, which could be mixed with the flare ingredients as a liquid, and allowed to polymerize after the flare was packed.

When a stoichiometric nitrate metal flare composition employing 200-mesh ingredients was tested, it was found to burn too rapidly for the intended purpose; hence diluents were incorporated to slow the combustion.[2] When oxygen-containing inert salts, such as carbonates or sulfate, replaced a portion of the nitrate, a decrease of the burning speed was satisfactorily effected.

A flare mixture, characterized as No. 47, was developed, and proper conditions for its use were fully investigated. The formula for No. 47 is:

Magnesium	16%
Aluminum	12%
Barium sulfate	40%
Barium nitrate	32%
Compounded with manganese dioxide	1%
Boiled linseed oil	8%

All solids 200 mesh or finer

This mixture, properly packed and cured, burned with a good light in as much as 300 ft of water, reached its peak candlepower within 3 or 4 sec after ignition, and burned with a steady flame if it was packed in a thin-walled casing. It formed a strong cake, so that thin-walled paperboard casings could be used without much danger of fracture of the cake by rough handling. If heavy casings must be used in the construction of a flare, barium sulfate should be replaced by barium carbonate, since flares containing the latter tend to form less clinker when burned in a confined space. The No. 47 flare had a burning speed of 10 sec per in., with a candlepower (4-in. diameter) of about 60,000 in water, or 250,000 in air.

Preliminary tests with underwater flares indicated that an object on the surface or under water was very well silhouetted by a 4-in. diameter flare burning in as much as 100 ft of water. On a number of occasions surface craft, tow targets, etc., were lighted up, and in one instance a school of fish in 40 to 50 ft of water was clearly visible when the flare was burning at 90 ft. The surface-illuminated circle was about 200 ft in diameter.

4.2.3 The Rocket Star

A need developed for a fast-burning star of very high intensity for use as a rocket head, and the standard Navy star was found unsatisfactory because of its tendency to develop peak candlepower slowly. A stoichiometric mixture of magnesium metal powder and sodium nitrate, both 200 mesh, bound with boiled linseed oil, was recommended as the most brilliant flare mixture available.

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[a] Aluminum (100-200 mesh), 9 per cent; barium nitrate, 58 per cent; magnesium (45-100 mesh), 27 per cent; paraffin wax, 6 per cent.

The flame of such a star was a deep yellow in color, and generated about 40,000 candlepower per square inch of burning surface. It burned at a rate of 0.2 in. per sec. If a white star with about the same characteristics is desired, the sodium nitrate should be replaced by barium nitrate.

4.2.4 Colored Flares

Colored flares were desired for use on the tail of radio-controlled bombs so that the bombs could be seen from the plane during the entire time of their descent. It was necessary that the flare and igniter function at altitudes up to 30,000 ft; that the flare have high light intensity so as to be plainly visible in full sunlight at the distances involved, and that it have a true color, so as not to be confused with ground lights or bombs dropped from other planes using a flare of a different color. This use required a burning time of about 1 minute with the size limited to 5 in. in diameter and 5 in. in length, including the ignition device. Colors requested were red, yellow, white, and green.

Flares which will operate under water will function at high altitudes, unless the low temperatures encountered affect their flammability. The high altitude aspect of the problem had, therefore, already been solved in the work with the underwater flare. The real difficulty lay in the development of clearly colored flames from formulas complying with the stoichiometric requirements for maximum efficiency in rarefied atmosphere.

Tests were made with yellow flares at a distance of 3,500 ft between observer and flare to determine what intensity was required. It was found that, even in full sunlight, the visibility and color recognition of the flare were entirely independent of candlepower but were functions of the apparent size of the flare flame. Even the glowing clinker of an extinguished flare could be seen at that distance, and the color was recognizable if the clinker was large enough to appear as more than a point of light.

Final tests were made on a range of 42,000 ft at sea level, and again it was demonstrated that if the flame was large enough to be seen as more than a point of light its color could be easily determined. The 42,000-ft distance was great enough for air absorption of light to play an important factor, and the colors were modified considerably by selective absorption; a clear yellow became deep orange, and grass green became lime green at that distance. There was, however, no question as to which flare was red, which green, and which yellow when three were burned side by side. It was found that the red flare, producing about 70,000 candlepower, was easily visible at 42,000 ft. A white flare rated at 140,000 candlepower was near the visibility limit, apparently because it burned with a much smaller flame.

It should be pointed out that the test condition, a 42,000-ft observing path at sea level, was extraordinarily severe since the air absorption was probably 50 to 100 times as great as it would be over a path from an altitude of 30,000 ft to earth. The greater air absorption should affect the apparent color of the flares, rather than apparent size and visibility.

The problem of attaining a colored flare of high color purity was not particularly difficult when red or yellow flares were sought but became increasingly so as the wavelength of the apparent color was made shorter. There were two reasons for this. First, the commonest impurities in heavy metal salts are sodium salts, which produce an extremely brilliant yellow flame on combustion. A small yellow component will not greatly affect the color purity of the red flare, as much as 5 per cent of a sodium salt being needed to make the flame appear orange rather than red. Mere traces of sodium salts will, on the other hand, completely destroy the green and blue flame colors, turning them to a sickly white.

Second, there is a continuous spectrum background to the line and band spectra that must be excited to produce strong color in a flame. The red flare-flame spectrum has a series of bands between 6,000 and 7,000 A wavelength, the yellow flare-flame spectrum has the sodium line at 5,900 A, and the green flare-flame spectrum has a band series between 5,000 and 5,500 A. Apparently a much greater thermal energy is required to excite a band at 5,000 A than is required for a band at 6,500 A, since the apparent black body temperature of the background spectrum increases markedly as the wavelength of the apparent flame color becomes shorter; thus, the background temperature of the red flame will be something like 1,700 K, while the green flame background temperature will approach 2,400 K. The total color effect of the background spectrum will be yellow to white and will vary in intensity as the fourth power of the temperature. There will thus be a much greater white background component for the green flare, as compared to the red, and the saturation and purity of the color will be of a much lower order. With the blue flare the background becomes so brilliant that it overpowers almost completely any blue band spectra that may appear.

The solution to the first problem is to use only the purest ingredients in the preparation of colored flares. The second problem can be solved only by devising some means of producing a truly monochromatic flare, which involves some new method of generating light energy other than the classic pyrotechnic devices. Pyrotechnic flares are probably near the limit of development, so that any further advance of major proportions may well have to be based on an entirely new principle.

The formulas recommended for use with the guided bombs were:[9]

	Red	Yellow	White	Green
Magnesium	12			16
Aluminum	9			
Mg-Al alloy (50-50)		36	28	
Manganese dioxide	1	1	1	1
Hexachloroethane	8			19
Strontium nitrate	20			
Strontium carbonate	50			
Sodium nitrate		45		
Sodium carbonate		18		
Barium carbonate			28	
Potassium oxalate			11	
Barium nitrate			32	55
Lactose				9

The mixed powders were compounded with the following amounts of boiled linseed oil: 5, 4, 3½, 2 per cent, respectively, for the four colors.

All these flares had saturated colors, were of sufficient intensity for daylight use, and had a burning speed of about 15 sec per in.

4.2.5 The Ignition of Star Mixtures and Flares

Since there was need for stars which would operate under unusual conditions, considerable thought was given to means of igniting such flares. A primer system was devised which appeared to be certain in operation. The second-fire primer was a stoichiometric mixture of 200-mesh magnesium and sodium nitrate, bound with boiled linseed oil; the first fire was 75 per cent second fire, mixed with 25 per cent of meal powder. This priming system, pressed on the top of a flare cake which is bound with linseed oil, will insure ignition from a gunpowder squib flash or any larger flame.

The actual mechanical ignition is of importance at high altitudes. A Bickford-type fuze will usually not burn at a pressure of 0.1 atmosphere, so any fuze train employed should be a pencil of a caked primer, such as that mentioned above. Mechanical match mixtures must operate at low temperatures, and it was found that the usual phosphorous lead-chlorate striker system could be used if a good quality glue was used as a binder, in place of the usual dextrin solution. All mechanisms must be designed for low-temperature operations and must allow for increased brittleness of the striking compounds. Two typical matches[9] which will operate at low temperatures and in a vacuum were recommended to Division 5 of the NDRC.

The problem of underwater ignition is somewhat different. It is necessary to get ignition of the primer well under way before the wetproof cover of the flare blows off. It was found that a tight, nonelastic seal would not work, for the high pressures developed over the primer from the ignition would produce an explosion violent enough to shatter the flare cake. The best answer seemed to be to use a plastic seal, such as might be obtained with a rubber dam or vinylite membrane over the flare cake.

[redacted]

4.3 THE PHOTOFLASH BOMB

4.3.1 Introduction

Most troop and supply movements near combat zones are made at night, and it is necessary to obtain clear reconnaissance photographs. The photoflash bomb produces the light for these photographs. The bomb is dropped from the reconnaissance plane and timed to explode about two-thirds of the distance to the ground. At the start of World War II, the bomb in use was a cylindrical cardboard case 6 in. in diameter, containing 25 pounds of flash powder consisting of 40 per cent potassium perchlorate, 34 per cent granular magnesium, and 26 per cent granular aluminum. It produced a peak candlepower of about 300×10^6. In order to obtain better ballistics, a streamlined steel outer case 0.06 in. thick was later used. This added confinement increased the peak candlepower to about 500×10^6, although the reason for the improvement was not apparent at the time. Because of the supply situation the formula was changed to 54.5 per cent barium nitrate and 45.5 per cent magnesium-aluminum alloy (50-50). This composition was also preferred because it was somewhat less sensitive than the perchlorate powder. The new powder produced about the same average intensity, but double peaks often occurred and the time to peak intensity was not consistent. Both of these last facts resulted in occasional bombs providing insufficient light during the time the camera shutter was open (0.01 sec starting about 0.015 sec after burst). This bomb was designated the M46.

4.3.2 The Explosive Photoflash Powder

A preliminary study was made with small (5 to 20 gram) flashes, testing all available oxidants, and it was concluded that potassium perchlorate was as satisfactory as anything available and practicable. In testing various metal powders, it was found that zirconium and titanium metals and their hydrides showed a marked catalytic effect on the speed of reaction of flash powders, and that the addition of 1 or 2 per cent of one of these catalysts to a mixture of aluminum and potassium perchlorate powders produced an exceedingly fast-burning powder. Without the catalyst the mixture was decidedly inferior as a flash producer.

Study of the powders, including the Army standard perchlorate powder, indicated that a stoichiometric mixture consisting of 38 per cent magnesium-aluminum alloy (50-50), with 62 per cent potassium perchlorate was far superior to the standard powder in thin-walled bomb casings such as that of the M46. With any powder, stoichiometric mixtures gave the greatest intensity and most consistent results. It soon became apparent that very little further improvement could be made in powder compositions and that any improvements would be made only by increasing the sensitivity of the powders. No recommendations were made concerning the titanium or zirconium catalyzed powders, because their extreme sensitivity made them practically laboratory curiosities.

4.3.3 Casings of Photoflash Bombs

Small-scale studies of perchlorate powders led to the belief that the effectiveness of flash powders depended largely upon the casings in which they were fired. A series of casings, ranging from paper bags through glass bottles to hand grenade blanks, provided a range of casing strengths, and it was found that the peak candlepower values increased in the casing series from paper, paperboard, and wood to a maximum with glass bottles, and fell off for the hand grenade blank.[3]

A series of test bombs with steel casings was prepared by Army Ordnance, and was test-fired at Aberdeen Proving Ground.[6] The casings ranged from 20 to 60 per cent of the total bomb weight of 50 pounds. The barium nitrate reached its maximum efficiency when the casing was 40 to 60 per cent of the total bomb weight, and fell off at 20 per cent, indicating that a true optimum casing powder weight ratio existed near the 50-50 point.

The perchlorate powder showed the best results in the lightest case (20 per cent of total weight), indicating that only little confinement was desirable for this powder. The results of these tests are tabulated below.

Powder Weight, lb.	Case Weight, lb.	Case Thickness	Candlepower at Peak
54.5 per cent Ba(NO$_3$)$_2$, 45.5 per cent Al-Mg alloy			
20	30	0.340 in.	1,300 $\times 10^6$
25	25	0.250	1,650 $\times 10^6$
30	20	0.182	1,650 $\times 10^6$
35	15	0.125	1,500 $\times 10^6$
40	10	0.077	1,350 $\times 10^6$
40 per cent KClO$_4$ 60 per cent Al-Mg alloy			
20	30	0.280	600 $\times 10^6$
25	25	0.210	600 $\times 10^6$
30	20	0.152	950 $\times 10^6$
35	15	0.109	1,100 $\times 10^6$
40	10	0.60	1,200 $\times 10^6$
62 per cent KClO$_4$, 38 per cent Al-Mg alloy (stoichiometric)			
20	30	0.280	950 $\times 10^6$
25	25	0.210	1,050 $\times 10^6$
30	20	0.152	1,600 $\times 10^6$
35	15	0.109	1,750 $\times 10^6$
40	10	0.060	2,000 $\times 10^6$

It was concluded that there is an optimum amount of confinement required for each type of flash powder for most efficient results.

Investigations were made of the faulty behavior of a number of experimental Army bomb models employing the barium nitrate powder in thin-walled bombs. Analysis of test-firing measurements and high-speed motion pictures indicated that even in the thin-walled bombs weak assembly points caused failures. The pictures showed, in some instances, bomb tails blowing off and bomb bodies flying away intact. After correction of the assembly faults much higher peak intensities and greater consistency were obtained.

4.3.4 Nonexplosive Flash Powders

The Germans used, and the British perfected, a photoflash bomb which was very much safer than the flash-powder bomb, and apparently was as effective.[11] The device consisted of a bomb casing with an axial burster of high explosive and the remaining space in the casing filled with flake aluminum powder. Magnesium was not effective. The weight of the burster as used by the British was one-third that of the aluminum. Detonation of the burster scattered the aluminum as a dust cloud and ignited it. The principal advantages of this type of bomb over the classical model were that it was less sensitive and that the oxygen was supplied by the air and need not be carried in the bomb. High-speed motion pictures of the bomb flash showed that the high-explosive burst blew the metal into a ring, and thus the flash had a central dark space. This may not have been true when aluminized HE bursters were used, which perhaps accounts for the better results obtained by the British with this type of burster. In addition, there was considerable doubt whether the metal dust was efficiently ignited, since the flash was usually highly irregular in shape, yet the dust cloud appeared to be symmetrical.

Only a moderate amount of work was done on this type of bomb, and considerable improvement should be possible. For example, the size and shape of the burster appear critical but have not been thoroughly investigated. More aluminum can perhaps be used by compressing it, with sulfur or other material as binder.

Aluminum-sulfur mixtures were investigated for use as an explosive, and preliminary trials were made of the mixtures as photoflash compositions. The best results were obtained with a mixture of 25 per cent flowers of sulfur and 75 per cent flake aluminum. When compressed at 5,000 psi, the mixture form a cake of specific gravity 1.85. A conical burster of about ¼ the diameter of the flash composition gave good dispersal and ignition, and showed better results than the cylindrical burster. An aluminized TNT burster was recommended. Only a small amount of work was done on this mixture and only tentative conclusions were drawn. The results indicated, however, that a rapid and high peak intensity was obtained.

It seems unlikely that either explosive or dust-flash bombs can be constructed in any feasible size to produce much greater than 2,000 $\times 10^6$ candlepower at peak, so further work must involve some fundamentally new method of producing a flash. One promising lead is the observation that the shock wave from a high-explosive detonation produces an extremely intense flash of very short duration when it is made to pass through an atmosphere of argon. A 200-gram explosive charge has been reported[4] to produce a peak intensity of 200 million candlepower. Preliminary tests[13] using RDX to produce shock-wave flashes were carried out just as the project was being concluded, with sufficiently promising results to justify further study of this technique.

4.4 INSTRUMENTS AND TECHNIQUES

4.4.1 Photographic Photometry

Most of the prewar flash photometry was carried out by means of a photographic method which is laborious and particularly liable to errors arising from a large number of sources. Photographic photometry is scarcely applicable to routine proof operations, but it is quite valuable for research and development, since it offers a check on the less arduous electronic methods of measurement. The check is valuable since the methods are completely different in principle, and are not subject to the same instrument errors.

The method is, briefly, the comparison of the blackness of images produced on a photographic film by the light to be measured and by some standard light of known intensity. Since, ideally, the blackness, or density, of the developed photographic image increases as the logarithm of the amount of light striking the emulsion, an error of density measurement will produce an anti-logarithmic error of light value, which will assume very large proportions unless density errors are kept small.

In actual practice, the ideal logarithmic relationship of density and amount of light is seldom attained; therefore the standardizing exposure should cover a wider illumination range than the flash to be measured. A standard graph of the amount of light versus density is plotted, and values of the unknown are read from the curve. The usual method is to place a sheet of film, which varies in transparency from about 90 to 0.1 per cent in a known manner, over the film to be standardized, and to impress the exposure from the standard lamp through the graded transparency. The standardized film strip will, on development, show a varying blackness corresponding to the varying transparency of the filter strip, or "step-tablet". From the known light output of the standard and the transparency of each step of the tablet, the amount of light producing each exposure on the standardized film is known, and the density-light intensity curve can be set up.

Care must be taken to use the same duration of exposure for standard light source and unknown value. The photographic emulsion does not respond equally to an intense exposure of short duration and a faint exposure of long duration, though both represent the same quantity of light. For example, an exposure of 0.01 foot-candle persisting for 10 sec will not necessarily produce the same density on the developed film as 100 foot-candles for 0.001 of a second, although both exposures are of 0.1 foot-candle-second of light.

Duplication of film processing with sufficient accuracy to make separate films photometrically comparable is not practicable; therefore each sheet of film should have its own standardization. Variations over a single sheet of film may reach large proportions unless extreme care is taken in the processing. It is necessary to develop the film at a constant temperature (a tolerance of ±0.1 C is recommended), fresh developer must be used for each film, and the film must be brushed throughout development with a camel's hair brush to insure uniformity. A good film-processing technique can only be attained by extended practice.

The latent image produced by the exposure varies in developability with time; hence, the standard wedge pattern should be impressed as soon as possible after the exposure of the film to the light to be measured. An error of as much as 25 per cent may be produced if this precaution is not heeded. There is some evidence that the temperature of the film at the time of exposure affects the results; although the magnitude of this effect is not well established, it is wise to maintain as uniform a film temperature during exposure as is practicable.

4.4.2 Photometry with the Oscillograph

The use of a photoelectric cell and oscillograph as a photometer results in a simple and accurate means of determining the light-producing characteristics of a flash. Satisfactory oscillographs are commercially available, and only a few precautions need be observed in their use. When a photoelectric cell is coupled directly, or through an amplifier, across the deflection plates of an oscillograph, a light pulse directed on the photocell will give rise to a voltage change in the circuit. This voltage change will produce a deflection of the electron beam of the oscillograph tube proportional to the magnitude of the change, which in turn is proportional to the intensity of the light pulse. If the deflection sensitivity of the oscillograph is calibrated in terms of a standard lamp, the intensity of the light source in candlepower can be determined.

The usual procedure of recording data is by some photographic method. The oscillograph can be made to sweep on a time axis perpendicular to the light intensity axis, either for a single sweep greater in duration than the period of the pulse, or in a continuous sawtooth sweep of short duration relative to that of the flash. In the first instance, a straightforward time-intensity curve will be photographed, while in the second a folded curve will be produced. A third recording method employs the oscillograph without a time sweep, and photographs the intensity sweep on a moving film to produce a time axis.

For accurate results the photocell must be of the electron emission type; the barrier-type cells react so slowly to a sharp light flash that they tend to act ballistically, and give a badly distorted frequency response, which results in a false time-intensity curve. Furthermore, the barrier cell is so insensitive that its output must be amplified, and obvious difficulties are encountered when the direct-current light cell is operated through an alternating-current amplifier.

The greatest advantage of the photographic technique of photometry is that the light unit is defined in terms of the spectral sensitivity of the photographic emulsion. If the oscillographic photometer is to be used, the spectral sensitivity of the photocell should be corrected to that of the photographic emulsion for accurate results in the study of light flashes to be used in photography. The correction can best be accomplished by means of color filters, although these filters are by no means easy to devise.

The cesium oxide photoelectric cell is probably the most satisfactory type available for use with the oscillograph in flash studies. Two photocells were used in this work, both cesium oxide emission cells of about the same spectral sensitivity. They were the RCA 919 and the Cetron CE-31-V cells, which showed peaks of sensitivity in the near ultraviolet and near infrared. A filter solution with high transmission between 6,400 and 4,000 A was used with these cells, causing them to correspond roughly in sensitivity to tri-X-panchromatic emulsion in the visible region. Extensive tests demonstrated that candlepower results, using the photographic photometer with tri-X-pan and the filter-photoelectric cell-oscillograph photometer, are comparable over a wide range of temperatures. The filter solution for the correction of these cells was used in a 1-cm glass cell. It had the following composition: Copper nitrate, cp $Cu(NO_3)_2 \cdot 3H_2O$, 60.4 grams, dissolved in 1 liter of a solution of methyl alcohol, cp absolute, 60 per cent by volume, and distilled water, 40 per cent by volume.

4.4.3 Spectrographic Studies of Flashes

Only preliminary spectrographic studies of flashes were made in the photoflash program, but enough was learned to point up some of the difficulties of such work. For spectrophotometric use the data must be of the highest precision, at the very limit of the present techniques of photographic photometry. For instance, color-temperature measurements by the spectrographic method are probably not better in precision and accuracy than ±3 per cent. The only other method at present in use employing color filters in front of photocells of two channels of an oscillograph is probably better when used to measure a light pulse with a completely continuous spectrum, but is not likely to be dependable for the study of flashes whose spectra contain lines and bands. The color filters are generally transparent over a 500 A wavelength range, and vary in transparency from point to point within that range, so that it is difficult to assign a mean wavelength to the filter for calculation purposes. The wide wave bands measured are likely to pass some of the discrete spectrum of the flash, which may lead to inaccurate results, since the calculations are based on a continuous black body spectrum.

An attractive scheme for temperature measurement is the use of a two-channel oscillograph coupled to a pair of photomultiplier tubes, which, in turn, are placed at the exit slits of two monochromators set to the desired wavelengths. The flash is picked up by the monochromators, and two time-intensity curves drawn on the oscillograph screen, one for each of the wavelengths desired for calculation.

For general studies of spectral distribution of flashes, the spectrograph seems to be the only feasible method if any detail of the spectra is of interest. A moving film spectrograph was used in this work.

4.4.4 High-Speed Photography

One of the most fruitful techniques for the study of photoflash bombs has been the high-speed motion picture camera. Cameras operating at a maximum of from 2,500 to 8,000 frames a second are commercially available, and are much more satisfactory for flash studies than for nonluminous objects. The ideal camera for flash work would operate at 10,000 frames per second, and would have a lens aperture variable from $f/2$ to $f/100$. The commercial 8,000-frame-per-second camera, running at full speed, and $f/16$ aperture gives pictures of flashes that are badly overexposed with black and white films. Color film with daylight sensitization is best suited to the work if optimum exposure is desired.

Frequently a flash will yield an abnormally shaped time-intensity curve, presumably the result of deviation from the usual nonspherical burst. High-speed motion pictures, especially when projected, often show what produced the abnormality. Faults discovered from time-intensity curves can often be corrected by a trial-and-error method, but the cost in time and materials is likely to be many times the investment in a high-speed camera if a full-scale bomb is involved.

4.4.5 Analysis of Results

Since the photoflash bomb is intended to generate an intense flash of light of short duration for photographic purposes, and since the photographic effectiveness of the flash depends upon its intensity and duration, the most important item of data is the time-intensity curve. The quality of a particular flash will be judged primarily by its peak candlepower, with total duration, total amount of light, the time elapsed between ignition and peak, and the efficiency as secondary criteria.

The U. S. Army Air Forces employ an aerial camera with a shutter speed of 0.01 sec for night photography. The shutter is actuated by a photocell mechanism which is tripped by the first light from the flash. A good flash should reach its peak intensity about the time the camera shutter is wide open, and since the time lag of the shutter mechanism is constant and reproducible, the flash bombs should, for best results, reach peak intensity within a millisecond or two of the open shutter time. Ideally, the flash should release the major part of its light within 0.03 sec after burst; any light produced after this time is of no use. In practice, full-scale flash bombs which function properly concentrate about half of the total light within the 0.03-sec period. The remaining half represents light produced at lower temperatures by natural cooling of the flash cloud. In a faulty flash, however, the flash powder may not be fully ignited at the burst, and may become productive in the dying period of the flash; such flashes are likely to show a marked diminution of peak candlepower.

The efficiency of a flash is usually expressed as the number of candlepower-seconds per gram of metal contained in the powder. Magnesium metal apparently will produce about 40,000 cps per gram under optimum conditions, but the usual figure for a full-scale flash bomb is 10,000 cps per gram. If the efficiency is much lower, especially if the flash is of abnormally long duration, it is safe to assume that the powder or bomb construction is faulty. Delayed peaks, multiple peaks separated by over 0.01 sec, or peaks with flat tops persisting for over 0.03 sec, usually occur when the bomb casing is of faulty construction. Frequently an otherwise normal flash will show a minor peak within 2 or 3 msec of the burst. This usually means that some of the ignited flash powder has blown out through the fuze before the bomb casing burst. The effect upon the quality of the flash is negligible, unless the tail blows off the bomb before the casing ruptures.

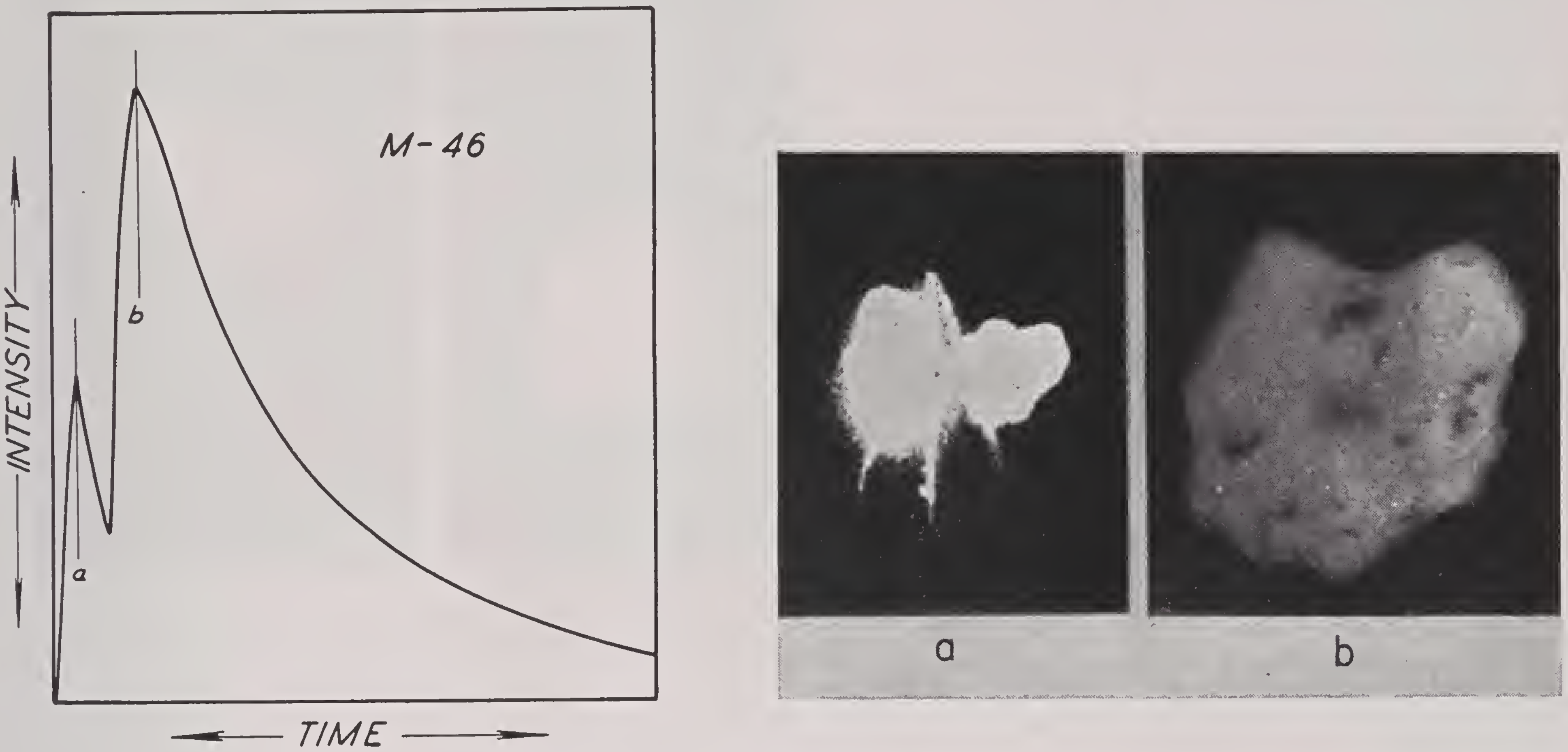

FIGURE 1. Flash A. M46 photoflash.

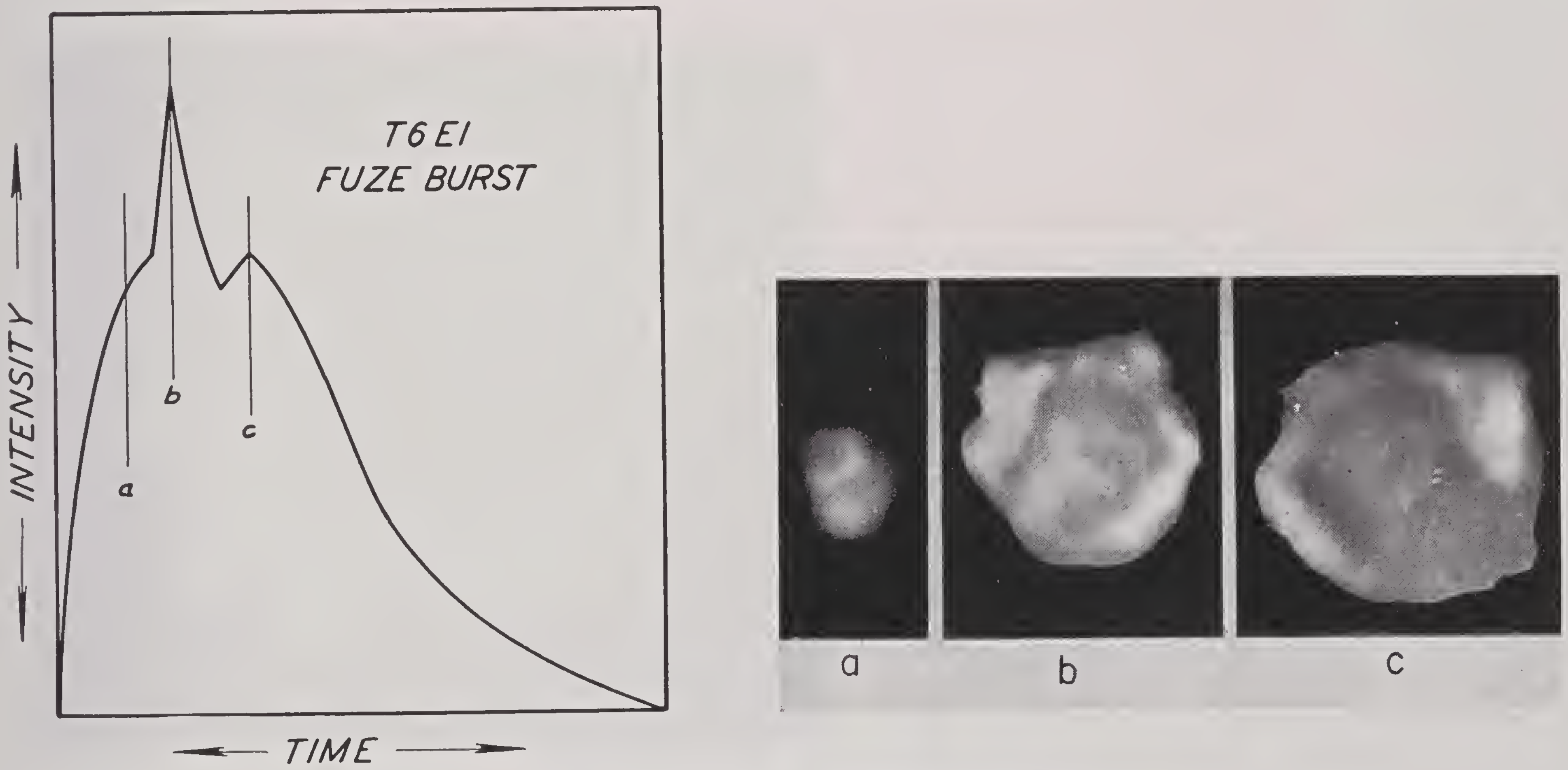

FIGURE 2. Flash B. T6E1 fuze burst.

High-speed motion pictures will usually confirm faults such as those mentioned above, especially if pictures are taken from both front

[redacted]

and side. If a fault is of a rarely encountered type, it is doubtful if the seat of trouble will be located without the aid of motion pictures.

The technique of analysis of results is illustrated by the accompanying Figures 1 to 4.

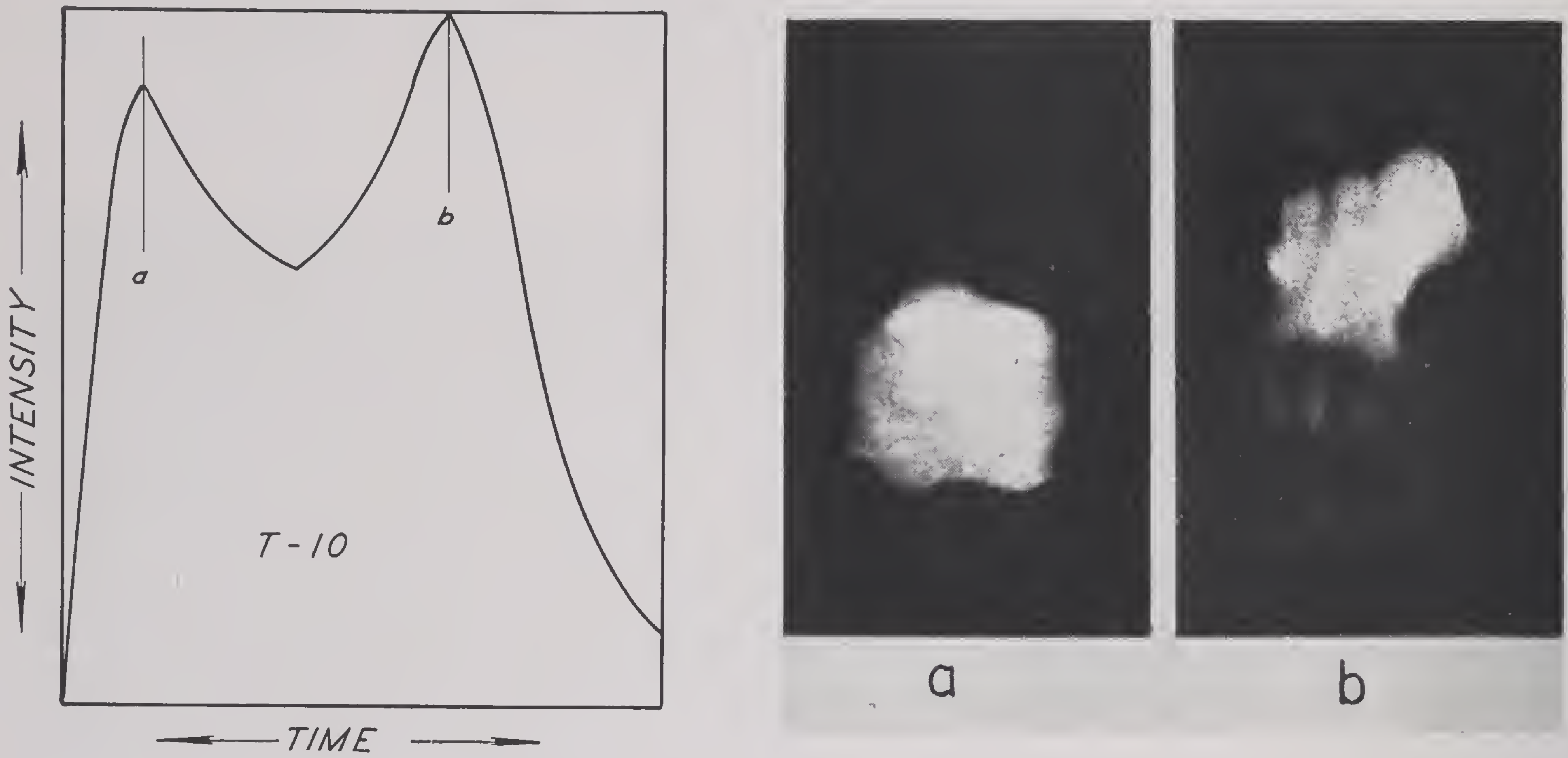

FIGURE 3. Flash C. T-10 flash cartridge.

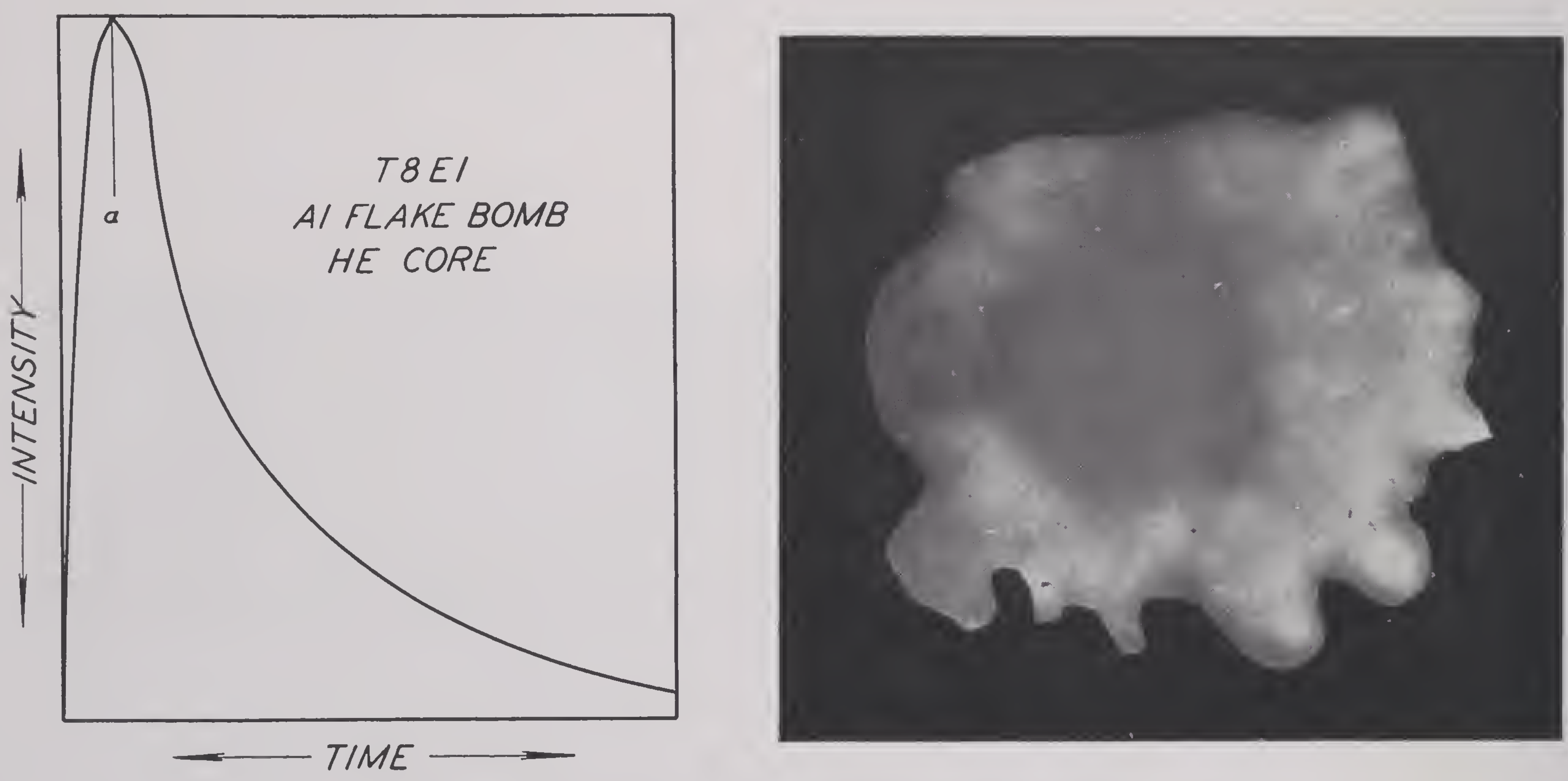

FIGURE 4. Flash D. T8E1 aluminum flake bomb.

FLASH A

The time-intensity curve is normal for explosive photoflash powder, except at *a* near the origin, where a minor secondary peak occurs. Such a condition is of frequent occurrence and appears to be caused by a flash through the nose fuze of the bomb (A-*a*). This has virtually no effect on the development or efficiency of the flash, although at times a dark center will be produced on the bright surface if the nose flash is not swallowed by the main burst (A-*b*).

Flash B

This flash is one of the most spectacular demonstrations of the effect of faults on record. A massive nose flash apparently occurred; although it does not show on the time-intensity diagram, it is clearly visible in the motion pictures. In addition, the tail appears to have blown off the bomb. A homogeneous ignition of the powder most certainly did not occur. There was, unfortunately, no motion picture record from the side of the flash, but a still picture shows two distinct bursts rather than the usual single globe. The burst at the nose developed rather slowly and was apparently fed with new material (B-*a*). The first-formed cloud was well cooled at the time of the delayed main burst (B-*b*), and produced a marked shadow over the flash surface. A third portion of the powder exploded still later (B-*c*), and still farther behind the main cloud. Such occurrences have been recorded only with the barium nitrate class of flash powder. This powder is a very good one when used in the proper bomb case but tends to display freakish behavior in inadequate cases. Apparently it is relatively slow-burning in the open but reacts with extraordinary violence when heavily confined.

Flash C

The T-10 flash cartridge was badly designed, having a light metal case with about 6 oz of flash powder and an extremely heavy-duty, high-explosive igniter. When fired, about half of the powder was nicely dispersed and ignited by the burster, and the remainder was not disturbed. When half of the cartridge was later ignited, probably by the flame of the first flash and not at the primary ignition, it took off in rocket fashion and finally burst to produce the secondary peak (C-*b*).

Flash D

In general, the safe type of flash bomb, in which a dust cloud of metal is dispersed and then ignited by HE, will yield a normal-appearing flash curve regardless of faults. Any improperly dispersed dust will probably not ignite, so only the primary flash will be noted. High-speed motion pictures show the characteristic dark center and irregular flash surface, which appears to be due to incomplete ignition rather than poor dispersal of the dust (D-*a*).

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Chapter 5

IMPROVED INFLATION OF LIFE RAFTS AT LOW TEMPERATURES

5.1 SUMMARY

Life rafts with the standard carbon dioxide inflation system, when cooled to −40 F, as can occur during high altitude flying, often required well over 15 minutes for proper inflation. By mixing nitrogen or nitrous oxide with the carbon dioxide, inflation could be obtained in slightly less than 15 minutes if the valve end of the cylinder was down. If it was up, no improvement occurred. In actual use the position of the cylinder would be a matter of chance. The use of compressed air in place of carbon dioxide gave very satisfactory inflation, but larger, and therefore heavier, cylinders were required, which made this solution unacceptable. Replacement of the carbon dioxide with nitrous oxide gave much improvement with the valve end of the cylinder down, full inflation occurring in 6 minutes. No improvement was obtained with the valve end of the cylinder up. However, by using a modified dip pipe in the cylinder with the nitrous oxide, the improved results were obtained regardless of the cylinder position. This combination of the use of nitrous oxide and the modified dip pipe appeared to be the most satisfactory method of altering existing equipment.

5.2 INTRODUCTION

Life rafts made of rubberized fabric were carried on all military planes flying over water. These rafts were carried folded and were inflated with carbon dioxide when they were needed. Several sizes of rafts and types of release from the plane were used, depending on the number of men in the crew and the construction of the plane. Fighter pilots wore the raft as a seat pack; in the bombers the raft was in a separate compartment from which it was generally ejected by the inflation after the carbon dioxide release was pulled, but in some planes the raft was ejected mechanically.

It was found that when the carbon dioxide was at a low temperature at the time of discharge, the raft was inflated very slowly, often requiring more than 15 minutes. This condition was serious in northern latitudes, since a man will generally not survive after immersion in cold water for this length of time. A plane is likely to sink within 30 sec of a crash landing, and if the ejection of the raft depends on the inflation, the slow inflation may result in loss of the raft.

5.3 CARBON DIOXIDE SYSTEM

If the cylinder was in an upright position (valve end up) when discharged, only gaseous carbon dioxide was emitted, leaving a large residue of solid carbon dioxide in the cylinder.[a] With rafts at −40 to −60 F inflated in air, only about 45 per cent of the cylinder contents were discharged in 1 minute. The remaining solid vaporized very slowly. When inflated in water, about 60 per cent of the contents of the cylinder was discharged. If the valve of the cylinder was down, the contents were discharged as liquid and the vaporization took place in the valve or manifold, where solid carbon dioxide was formed, usually plugging the system and stopping the inflation within a few seconds of the time the valve was opened. Practically no inflation was obtained for several minutes.

The life raft inflation system used a cylinder capable of withstanding an internal pressure of approximately 3,000 psi. This cylinder was usually charged to a density of 42.5 pounds of carbon dioxide per cubic foot of inside cylinder volume. At this density, 99.2 per cent by weight of the carbon dioxide was liquid at −65 F. The ratio of liquid to gaseous carbon dioxide progressively decreased as the temperature increased, and became zero at the critical temperature of 88.4 F.

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[a] Some cylinders were equipped with a rigid dip pipe extending to the bottom of the cylinder. Thus liquid discharge occurred when the valve end was up, and gaseous discharge when the valve end was down. Other cylinders were equipped with a short flexible dip pipe which gave liquid discharge both when the cylinder was horizontal and the valve end of the cylinder was down. As an aid to the visualization of the problem, the case of the simple cylinder with no dip pipe is used as an example in discussing relations applicable to all three cases.

The heat required to convert the carbon dioxide from liquid to gas came from several sources: the sensible heat obtained by cooling liquid carbon dioxide, the cylinder, raft and surroundings; or from the latent heat of fusion liberated when the carbon dioxide solidified. When a cylinder at room temperature was discharged, the sensible heat of the liquid carbon dioxide and of the cylinder was sufficient to vaporize nearly all the carbon dioxide, so that only a small remainder froze. However, if the cylinder was initially at low temperature, the heat necessary to vaporize the carbon dioxide was almost entirely derived from the heat liberated by the solidification of a large fraction of the carbon dioxide. This resulted in only partial vaporization and only partial inflation of the raft.

Whether the evaporation of liquid carbon dioxide at low temperatures occurs in the cylinder or in the raft, a substantial quantity of the carbon dioxide will be solidified. In order to vaporize the solid, heat must be transferred from the surroundings to the residual material, and this process is very slow. The rate at which heat is transferred depends on three factors: the driving force or temperature difference, the area available for heat transfer, and the resistance to heat transfer per unit area. Applying these concepts to the problem of securing the vaporization of carbon dioxide, several conclusions are apparent. The rate of vaporization of carbon dioxide will be higher if the snow is in the raft rather than in the cylinder, due to the larger area over which the snow may be scattered within the raft. Since resistance to heat transfer from air is roughly ten times as large as that from water, the snow will vaporize much faster if the raft is in the water. The resistance to heat transfer to a boiling liquid is much lower than that offered to sublimation of a light powder substance such as carbon dioxide snow. Hence, heat will be transferred to the liquid carbon dioxide much faster during the period of initial vaporization than at any later time. The temperature of the surroundings will not greatly affect the rate of heat transfer, since the temperature difference is 180 F and 138 F at ambient temperatures of 70 F and 28 F. However, under the most favorable conditions for heat transfer with snow inside the raft and the raft in the water, the time required for vaporizing the residual carbon dioxide snow will be measured in minutes and not seconds.

5.4 PROPOSED MODIFICATIONS

From this preliminary examination of the problem, it was apparent that the carbon dioxide system was unsatisfactory: (1) because sufficient heat was not available for vaporizing all liquid carbon dioxide, (2) because of passage plugging by solid carbon dioxide, and (3) because of the low rate of sublimation of the residual solid formed during vaporization. The above consideration suggested several methods of improving operation.

1. Use of a material in place of carbon dioxide that would not be liquefied at −65 F, such as dry-compressed air, thus eliminating the necessity of supplying latent heat and removing the possibility of plugging of passages by solid.
2. Use of material in place of carbon dioxide, such as nitrous oxide, which would not solidify under the conditions encountered, because its freezing point was below its boiling point at 1 atmosphere pressure.
3. Use of a material such as a nitrous oxide-carbon dioxide mixture having a lower freezing point than that of carbon dioxide, thus permitting a larger amount of vaporization in the valve-manifold system before solid formation occurred.
4. Use of mixtures of a noncondensable gas, such as air or nitrogen, with liquid carbon dioxide. This should give a greater initial discharge from equipment at low temperature.
5. Redesign of the valve-manifold system in order to reduce the amount of vaporization occurring therein.

6. Redesign of the cylinder dip pipe in order to secure liquid discharge regardless of cylinder position, thus ensuring vaporization in the raft rather than in the cylinder.

7. Storage of life rafts in heated compartments so that they did not reach the low temperatures of the air at high altitudes.

8. Supply of heat to the cylinder by special means, such as burning powder or some other chemical reaction involving heat.

5.4.1 Permanent Gas

The use of a cylinder charged with compressed air gave the best inflation observed, but had the disadvantage that a larger and heavier cylinder was required. A Navy Mark I raft, equipped with a Mark IV cylinder containing compressed air, gave a 90 per cent inflation in 15 sec with the raft initially at −40 F. The normal cylinder for this raft weighed 3.3 pounds and contained 1.3 additional pounds of carbon dioxide; the larger air cylinder weighed 7.2 pounds and contained 0.87 additional pound of air. This added weight was considered too great a handicap.

5.4.2 Nitrous Oxide

Nitrous oxide (N_2O), substituted for the carbon dioxide in equal weight, gave the same volume of gas. When this gas was discharged from a cylinder with the valve up (gaseous discharge), the results were no better than with carbon dioxide. A large portion of the material remained as a liquid in the cylinder, cooled to its boiling point at atmospheric pressure. With the valve of the cylinder down (liquid discharge), no plugging of valve or manifold occurred because no solid was formed. The cylinder was completely emptied in a maximum of 13 sec. Unvaporized liquid, however, was deposited in the manifold and raft, so that the degree of inflation of the raft in 1 minute was not appreciably better than with gaseous discharge of carbon dioxide. However, heat transfer to a liquid (nitrous oxide) is much greater than to a solid (carbon dioxide), and this advantage was further increased by the greater area covered with the material in the raft rather than in the cylinder. As a result, the raft, originally at −40 F, was 100 per cent inflated in air in about 6 minutes.

5.4.3 Mixtures of Nitrous Oxide and Carbon Dioxide

Mixtures of nitrous oxide and carbon dioxide ranging from 6 to 83 per cent nitrous oxide were tested. The results obtained were no better than for carbon dioxide alone, when gaseous discharge was used. With liquid discharge, however, there was no plugging of the system with solid if 13 per cent, or higher, nitrous oxide was used.

5.4.4 Mixtures of Permanent Gas and Carbon Dioxide

The addition of nitrogen to the standard carbon dioxide charge in sufficient quantity to increase the pressure by 400 to 800 psi gave better results than the addition of nitrous oxide. Gaseous discharge was faster than for carbon dioxide alone, although about 40 per cent was left as solid in the cylinder, as with carbon dioxide alone. No plugging occurred during liquid discharge.

5.4.5 Redesign of Valve Manifold System

In the standard inflation system the carbon dioxide passed from the cylinder through a valve into a distributor, from which it passed through eight ¼-in. holes into the raft. These holes were large enough so that considerable expansion, resulting in vaporization, took place in the manifold, with consequent solidification of part of the liquid. If vaporization occurred in the valve manifold system, the resulting solid usually plugged the valve or manifold and prevented the discharge of the cylinder until enough heat was transferred to the plugging solid to vaporize it. The amount of vaporization occurring in the valve manifold system was determined by the relative dimensions of the passages. Thus, if a small passage in the system was followed by a larger opening, the material flowing had to have a larger specific volume at the second point, since the weight passing the two points was necessarily equal. This larger specific volume was obtained by vaporization. If the heat necessary for the vaporization was not available from the sensible heat of the liquid carbon dioxide, solid formation occurred with probable plugging. By reducing the size of the distributor openings, the expansion in the distributor was reduced and the solid formation and plugging eliminated or materially reduced.

With the standard system, liquid carbon dioxide, if at a low temperature, could not be discharged from the cylinder because of plugging. Using $\frac{1}{16}$-in. holes in place of the $\frac{1}{4}$-in. holes, the cylinder contents were discharged through the manifold as a liquid in 20 sec. Solid carbon dioxide formed in the raft, however, and inflation was not complete until this solid was vaporized. A Navy 4-man raft was tested with this modified distributor, with the raft cooled to −53 F and inflated in water at 38 F, using a liquid discharge. The raft was 40 per cent inflated in one minute, 80 per cent in 7 minutes, and 98 per cent in 14 minutes. Although this result was considerably better than would have been obtained with the standard distributor, it was not satisfactory.

Distribution of carbon dioxide or nitrous oxide inside the raft was improved by replacing the metal distributor caps with long flexible tubes containing a large number of properly distributed small holes. Using these tubes, 80 per cent inflation was obtained with carbon dioxide or nitrous oxide in air in less than three min or in cold water in 45 sec. These tubes, however, add weight and might crack with continued use or might burst when operated at high temperature.

5.4.6 Redesign of Cylinder Dip Pipe

In operation of standard inflation equipment, either gas or liquid discharge could be obtained depending on the position of the cylinder. As has been explained, liquid discharge produces much better results than gas discharge with either carbon dioxide or nitrous oxide. In order to ensure a rapid emptying of the cylinder with the valve end either up or down, an improved dip pipe was designed (Figure 1). This consisted of a rigid pipe *B* extending from the valve end of the bottle to a point near the opposite end. A flexible weighted tip *C* was attached to the open end of the rigid pipe

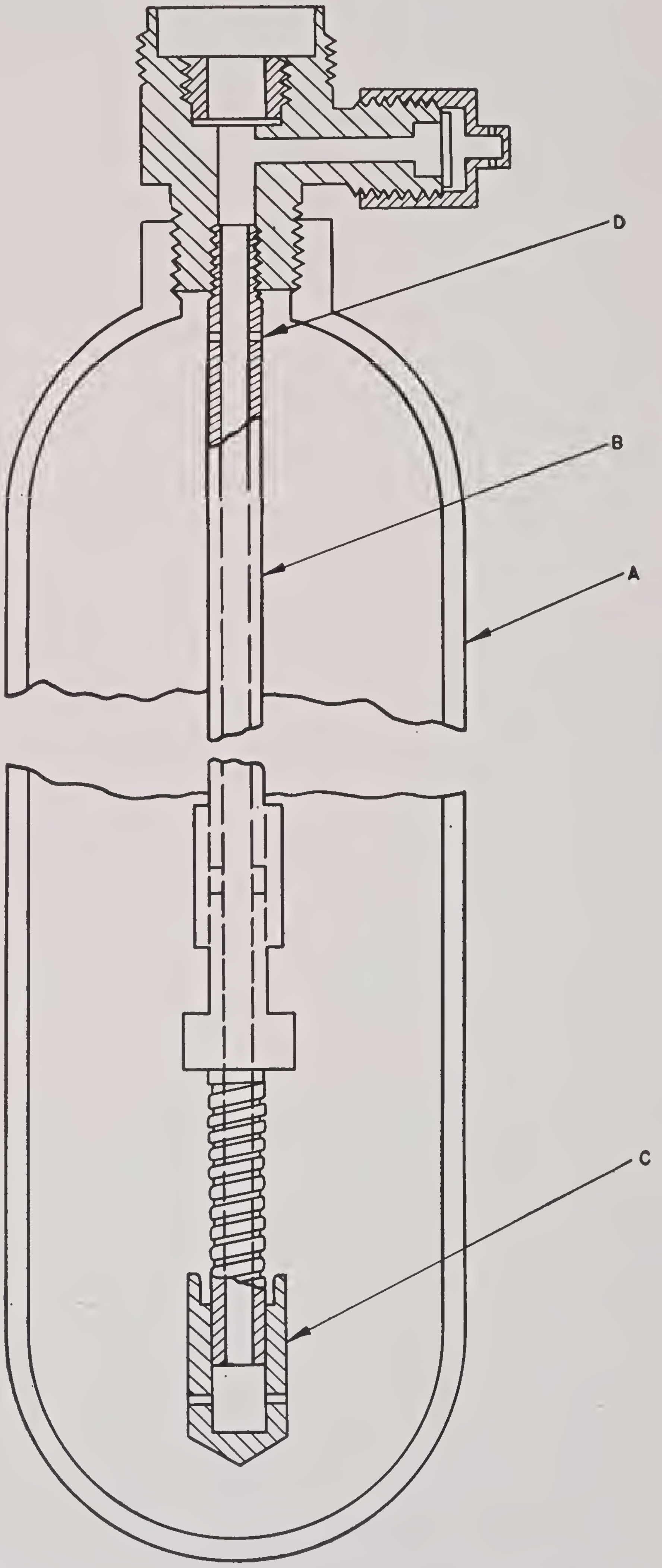


FIGURE 1. Improved dip pipe for gas bottle used to inflate life-rafts.

and 1 or 2 holes *D* were drilled in the wall of the rigid part of the dip pipe near the valve end of the bottle. Using this dip pipe with the valve end of the cylinder down, liquid flowed from the cylinder through the holes *D* near the valve end. With the valve end up, the liquid was forced through the holes in the weighted tip by the gas pressure above the liquid.

With this improved dip pipe, results with nitrous oxide were very much improved. At least 95 per cent of the material was discharged into the raft within 30 sec regardless of whether the valve was up or down. Results with carbon dioxide were not as successful. Enough vaporization still occurred to convert up to 60 per cent of the material to solid, which remained in the cylinder or manifold.

5.4.7 Storage in Heated Compartments

Heating of the compartment of the plane in which the rafts were stored had been considered by the Army and turned down because the electrical system was already overtaxed and heat by engine exhaust gases was not considered safe on account of the danger of carbon monoxide entering the crew's compartment as a result of leaks.

5.4.8 Supply of Chemical Heat to Cylinder

It would probably be possible to supply heat to the cylinder at the time of inflation by burning gunpowder or by other chemical reaction. Inflation could also probably be obtained by the products of combustion of burning powder or by gas generated by chemical reaction, such as metal hydride and water. These systems, however, would involve complete redesign of the inflation equipment and would not have been applicable to the thousands of units already in use, and for this reason they were not investigated.

The development of a chemically heated carbon dioxide or nitrous oxide cylinder would be a logical subject of a future investigation.

Chapter 6

ADVANCED POSITION IDENTIFICATION

6.1 SUMMARY

As a temporary marker for advanced positions in the jungle, a paste was developed which would change from white to leaf green in about 1 hour. The composition was based on the use of barium sulfate as a pigment plus the dye Green AB in the stabilized leuco form. Although this material seemed suitable for distribution from a small airplane, the amount needed was more than the limit of about 2 pounds stipulated for use by ground troops.

6.2 INTRODUCTION

In combat operations, there was frequent difficulty in knowing where the front lines were with sufficient accuracy for direction of artillery fire and bombing from airplanes. This was particularly true in jungle operations where colored cloths laid down by the ground troops could not be seen from the air and where smoke signals did not rise straight up, but diffused through the trees, making the source difficult to estimate. Even in open country, smoke drifts too much in any wind, and both smoke and flares have too short a life to be sure that they will not be gone before they are sighted. Many casualties were inflicted on our own men because of inability to tell exactly where they were.

6.3 VANISHING MARKERS

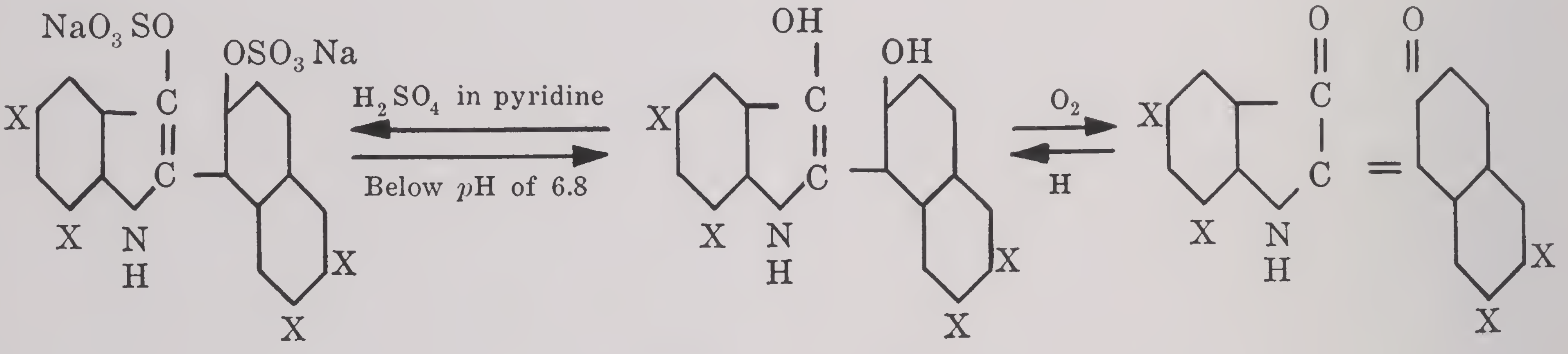
A possible solution to this problem was a material which could be used for marking the foliage and would disappear after a short time (¾ to 1½ hours). It was thought that such a marker could be placed on the foliage by the ground troops with existing munitions, such as mortars or rifle grenades set to burst above treetop level, or that it could be sprayed onto the trees by a slow-flying observation-type plane. A plane of this type could pick up signals from the ground where fast, high-flying planes could not.

Previous work had shown that visibility depends more upon brightness contrast than upon color contrast. Since the reflectance of foliage is very low (about 10 per cent), a white or light color of high reflectance should produce the greatest visibility against a background of leaves. The color is of minor importance as long as the reflectance is high enough to give a large brightness contrast.

Probably the best way to obtain the initial brightness would be to use a dye originally light-colored or colorless and depend on a pigment or substrate for the high reflectance. For best results the substrate should be an opaque white with good covering power. Barium sulfate was used for this work, although it is possible that better results could be obtained with one of the following: calcium carbonate, alumina hydrate, zinc oxide, magnesium carbonate, or titanium oxide.

A liquid mixture for this purpose must be of a proper viscosity for efficient distribution, have a maximum stability of suspension, and maximum covering power. To obtain maximum covering power, it is necessary to use as large a solid-liquid ratio as possible. The proper viscosity depends upon the method of distribution and can be determined only by experiment. If the suspension is not stable at the solid-liquid ratio established by the preceding requirements, it can probably be made so by the addition of stabilizing materials. Six hundred grams of barium sulfate to 1 liter of liquid gave a stable suspension of not too high viscosity, and was used as the ratio in this investigation. The powdered barium sulfate was mixed with the liquid in a high-speed Eppenbach-Homorod mixer. In a column of this liquid 15 cm high, the barium sulfate showed no settling after 5 hours, had settled 0.2 cm after 24 hours, and 0.2 cm after 336 hours.

The process of changing the color of the paste by exposure to the air can depend upon oxidation. There was only one general class of dyestuff which had as the final step in its preparation the oxidation of a substance (leuco form), which was colorless, or white, or lightly colored. This was the indigoid type of vat dyestuffs which consisted of derivatives of indigo and thioindigo. The indigo derivatives may be blue, yellow, or green, and the thioindigo derivatives are generally red. Since the colorless or leuco form was easily oxidized and therefore quite unstable, a stabilized form (indigoid, solvat, algosol) of the leuco had been developed. The relationship between these three forms, stabilized leuco, leuco, and dye of this class of dyestuffs, is shown below for Green AB:



(X represents any halogen)

Green AB

Table 1. Time for the development of color of dyes with 5.3–6.0 *p*H.

Dye	Vendor	*p*H	Time for color change (open shade on sunny day)
Pink IREX	Carbic Color and Chemical	6.0	8 min
Pink R	National Aniline	5.7	8 min
Orange R	National Aniline	5.7	8 min
Yellow CG	National Aniline	5.7	10 hr
Yellow GK	Carbic Color & Chem.; Gen'l Dyestuff	6.0	10 hr
Green AB	Carbic Color & Chem.; Gen'l Dyestuff	5.5	70 min
Blue O	National Aniline	5.7	25 min
Blue 4B	National Aniline	5.5	25 min
Blue 6B	National Aniline	5.3	20 min
Blue O4B	Carbic Color & Chem.; Gen'l Dyestuff	5.6	10 min
Red violet RH	National Aniline	5.7	8 min

It is apparent that for the stabilized form to be oxidized it must first be made acid. Acid must therefore be added to the paste compositions.

Pastes were made of the available stabilized leuco forms of dyes, and tests made of the time for the change of color to take place after exposure to the atmosphere. The paste formula was: barium sulfate—60 grams, water—90 ml, 6N sulfuric acid—10 ml, and stabilized leuco—1.5 grams.

The paste was spread in a thin film on white paper and the time taken from the initial spreading until a color of medium strength had formed. This time was probably somewhat longer than it would take for the paste to become invisible from the air. The results are tabulated in Table 1.

Green AB was the only pure dye found to give a color change within the required time limit. However, there was an indication that it might also be possible to develop a blend of one of the blue indigoid vat dyes and a yellow pigment or dye which would give a color change within the required time limits.

All dyes of the triphenylmethane, methylene blue, and safranine groups are also capable of yielding leuco compounds. As in the case of the anthraquinone and sulfurized vat dyestuffs and in contrast to the indigoid vat dyestuffs, the leuco compounds of these dyes are generally somewhat colored. However, the leuco compounds of a few dyes in these other groups are colorless or lightly enough colored to be of use. An example of this in the triphenylmethane series is malachite green. There are undoubtedly other examples.

It was noticed that as the paste dried, the color change process was retarded and the final developed color was light and very spotty. To overcome this, sufficient glycerin was added to the paste to keep it moist for several hours of exposure. This added glycerin had no effect on suspension stability. A more uniform and complete color development was thus obtained. The weight ratio of glycerin to water required is between 15/85 and 30/70. A 30/70 ratio was recommended. In paste compositions a volume-for-volume replacement is made of water by glycerin-water solution. Triethanolamine and diethylene glycol were tried in place of glycerin. Although neither was as satisfactory as glycerin, diethylene glycol could be substituted if necessary. The formula recommended for use, therefore, was:

Barium sulfate	60 g
Water	63 ml
Glycerin	27 ml
6N Sulfuric acid	10 ml
Stabilized leuco form of Green AB	1.5 g

In order to estimate the contrast that this paste would have against a forest background, reflection measurements shown below were made of thin layers of the paste on a maple leaf background at different development times.

Development Time of Paste (min)	Reflectance (per cent)
0	55
15	42
30	35
45	20
60	16
75	11
Green leaf background	10

The light source of the filter-type photoelectric spectrophotometer made by the Photovolt Corporation gave a wavelength band of 4,000 to 7,500 A and is designed to give a measure of brightness. The pastes were exposed to direct sunlight between 10:00 and 11:00 a.m. EWT, in the month of October. After 75 minutes, the reflectance contrast between the paste and the leaf green background is negligible. If the developed color of the paste is a close match for the leaf green, then the paste would be invisible even at close range.

A rough determination of the covering power of these pastes was made by spraying with a compressed air-spray gun on a surface of pebbles and dirt on an asphalt roof. A uniform and continuous covering was obtained over an area of 45 sq ft with 800 ml of barium sulfate slurry made up in the ratio of 450 grams of barium sulfate to 1 liter of water. It is realized that the use of a spray gun resulted in a much more efficient distribution than can be obtained with any distributing mechanism used by the Services. Allowing a safety factor of 2 and considering the facts that the paste used in the experiment contained 25 per cent less barium sulfate than any slurry previously studied (600 grams barium sulfate to 1 liter of liquid) and that there was an estimated 5 to 10 per cent spray lost in the above experiment, it seems safe to assume that 1 quart, or 3.0 pounds, of a paste containing 60 grams solids per 100 ml liquid will cover an area of 30 sq ft; 0.62 gallon, or 7.5 pounds, a circle 10 ft in diameter; and 2.5 gallons, or 30 pounds, a circle 20 ft in diameter.

These weights show that the paste-type marker would be too heavy for use by the infantry. However, it was felt that the color-changing paste discussed above might be usable by distribution from aircraft. No tests of distribution from the air or visibility of the mark from the air were made. Pilots have stated that a white panel 10 ft in diameter can be easily seen from an altitude of 2,000 ft if its position is fairly well known beforehand. For use as a position marker it has been assumed that an area about 20 ft in diameter would be required.

Powders involving the same type of color change were tried, but were unsuccessful because of greater difficulty in obtaining adequate timing of the color change and because a dry powder did not stick to the foliage. Tests were made with deliquescent powders (calcium chloride and lithium bromide), but it was found that the time required for the salt to pick up sufficient water to dissolve and disappear varied greatly with temperature, humidity, and particle size. This is shown in the following table giving results with calcium chloride.

Particle size	Relative Humidity at 31 C		
	30%	50%	90%
12 mesh	150 min	45 min	30 min
20 mesh	30 min	20 min	8 min
Fine crystallized	9 to 10 min	6 to 7 min	4 to 5 min

It is probable that the resulting concentrated salt solutions would kill the foliage and leave a permanent mark.

Chemicals which would disappear by volatilization were investigated. Of all commercially available ammonium compounds only ammonium carbonate gave favorable results, and this material killed all foliage with which it came in contact within 24 hours. The organic chemicals tested were naphthalene, *p*-dichlorobenzene, and camphor, none of which were satisfactory.

6.4 PARACHUTE MARKERS

Any further work on this project should include an investigation of the use of parachutes as markers. It is possible that they would be light enough to be carried by ground troops. There are two possible methods of providing for the disappearance of the parachute. One would be by impregnation with the leuco form of a green vat dye, so that the white parachute would turn to dull green in 1 to 2 hours. The other possibility would be the use of a highly inflammable paper, striped or crisscrossed with explosive material contained within waterproof tape, and having attached to it a time detonator to set off the explosive after a given time. If used by the ground forces, a projectile containing the parachute could be shot into the air and at a predetermined height the parachute would be ejected from the case by a powder charge. The case could remain attached to the parachute, acting as a weight to pull the parachute rapidly to the treetops.

The weight needed per mark seems definitely to favor the parachute type of marker over the color-changing powder or paste. A smaller mark would be needed than with powder or paste because of the solid continuous nature of the mark obtained with a parachute as compared with a spotty mark from pastes or powders. Calculations based on the weight of currently used paper parachutes show that 1 pound of paper will give a mark 10 ft in diameter. Including the weight of the case and powder charges, a parachute marker projectile for a 10-ft diameter mark might weigh 3 pounds and have an internal volume of about 3 pints.

Chapter 7

CHEMICALS FOR GENERATION OF HYDROGEN

7.1 SUMMARY

Several chemical sources of hydrogen were investigated as possible improvements on the ferrosilicon-caustic system for filling meteorological balloons. The addition of water to lithium hydride was known to be a convenient and efficient method, but lithium was very expensive and in short supply. Accordingly, a method of making relatively cheap lithium was worked out. This involved calcining spodumene with high-calcium lime, leaching, settling, and decanting or filtering. The filtrate, containing finely divided calcium hydroxide, was treated with sulfur dioxide, filtered, and low-cost lithium hydroxide obtained by evaporating and drying of the filtrate. The lithium hydroxide was reduced at low pressure and at 1100 C by ferrosilicon in the presence of lime and lithium metal distilled off and recovered with high yield. The estimated cost of lithium hydride produced by this process was $3.00 per pound as compared with $12.00 per pound by the older electrolytic process.

A briquetted mixture of sodium hydride and aluminum was developed as a much cheaper source of hydrogen. This material had many advantages, although the volume of hydrogen per pound of chemical was only about 40 per cent of that from lithium hydride. Cheap and simple generators with the sodium hydride-aluminum mixture for field use were developed for inflating small balloons.

The possibilities of lithium and sodium borohydrides were investigated, but the manufacture of these materials appeared to be too complicated and expensive for the purpose.

7.2 INTRODUCTION[1]

The Signal Corps employed the reaction of ferrosilicon and sodium hydroxide for the generation of hydrogen in the field for filling meteorological balloons. The generator used produced the gas at 2,000 psi and was of heavy construction (450 pounds uncharged). The reaction of the chemicals was not complete; therefore the charge of 9.5 pounds of solids produced only 80 to 90 cu ft of hydrogen. Besides the weights of equipment and chemicals, a further disadvantage was the difficulty of removing the residue from the generator after use; the sodium silicate residue solidified on cooling, and large amounts of hot water were required for cleaning. It was desired to obtain a generator for field use which was lighter in weight, easier to operate and which required the shipment of less generating chemical.

A number of chemicals were considered, the more promising of which are listed below.

	Cu ft of Hydrogen from 1 Pound of Chemical
Ferrosilicon and caustic soda (actual)	9.5
Sodium hydride (theoretical)	15.
Sodium hydride and oil (actual)	16.
Sodium hydride and aluminum (actual)	16.
Calcium hydride (theoretical)	17.
Calcium hydride commercial (actual)	7.
Lithium hydride (actual)	42.
Sodium borohydride (theoretical)	38.
Sodium borohydride (actual)	16.
Lithium borohydride (theoretical)	65.

The listed chemicals, except ferrosilicon, depend only upon the addition of water for the production of hydrogen.

Lithium hydride and ferrosilicon were the only materials commercially available. The lithium hydride was used on life rafts for producing hydrogen to fill balloons which carried up an antenna for an emergency radio transmitter. However, the available supply was not sufficient for meteorological balloons, and the price ($12.00 per pound) was considered too high for this use. The material was considered satisfactory from an operations standpoint.

The NDRC investigation consisted primarily of two phases: the development of a new and cheaper method of producing lithium hydride, and the development of a method of manufacturing a sodium hydride-aluminum mixture. The second phase included the design and production of experimental generators for use with the new hydride-aluminum mixture.

7.3 PRODUCTION OF LITHIUM[3,8]

The method used for producing lithium metal was the electrolysis of fused lithium chloride. This was expensive because of the high cost of the electrolysis operation, the necessity of using a high purity salt, and the low concentration of lithium in the ores. The principal lithium ore is spodumene, a complex mineral composed principally of lithium-aluminum silicate, with varying amounts of sodium and potassium, and containing up to about 3 per cent lithium.

The new process which was developed for lithium production depended on reduction under vacuum of lithium ores or salts and separation of the metal by distillation. Ferrosilicon was used as the reducing agent. Results indicated that the metal, or hydride, could be produced at one-third or less of the cost by the electrolytic method.

Lithium has a remarkable affinity for oxygen and, at higher temperatures, for other gases such as carbon dioxide, carbon monoxide, nitrogen, hydrogen, and water vapor. Because of the rarity and high cost of the inert gases, such as argon and helium, the best means for the exclusion of reactive furnace gases is by vacuum. The use of vacuum also permits the distillation of lithium from the reaction charge in a fairly pure state, and the removal of lithium from the reacting charge drives the reaction in the desired direction.

The production of lithium was attempted by reduction of spodumene and of the three principal lithium salts, the chloride, carbonate, and hydroxide. The reducing agents investigated were ferrosilicon and finely divided metallic aluminum, finely divided metallic magnesium, finely divided metallic iron, and calcium carbide. Ferrosilicon was found to be the most efficient.

It was not possible to produce lithium by vacuum thermal reduction of the chloride because of the high vapor pressure of this salt; the chloride volatilized out of the reaction zone before reduction could take place. Lithium could be produced from the carbonate using ferrosilicon and lime, but this reaction had several disadvantages. The carbon dioxide and carbon monoxide formed required the additional pumping of 2,000 cu ft of gas per pound of lithium at a pressure of 10 mm. Also, the reduction of the carbonate formed some lithium carbide which contaminated the product, reducing the purity to 80 per cent and causing the product to be pyrophoric. Lithium was produced satisfactorily by reduction of spodumene with ferrosilicon in the presence of lime, but the concentration of lithium in the charge was only 0.9 per cent, necessitating the handling of large quantities of material in the vacuum retorts. The hydroxide gave the best results. Because of the high cost of the commercially available lithium hydroxide, which was produced from the chloride, a process was developed for obtaining the hydroxide directly from spodumene.

The process finally recommended was as follows: a mixture of 30 per cent spodumene and 70 per cent high-calcium burned lime was calcined at 1050 C for 1 hour. The calcined material was leached with water just under 100 C, the slurry was settled, and the liquid decanted. Besides lithium hydroxide, the liquid contained a large quantity of lime, slaked during the leaching to produce a milk of lime suspension that was not amenable to thickening and filtering treatment by ordinary means. By treating this suspension with sulfur dioxide gas to the extent of 7 per cent of the amount necessary to react with the lime, a slurry was produced which did not settle readily, but which could be easily filtered. This enabled easy separation of the lithium hydroxide solution from the calcium hydroxide. Evaporation of the solution and drying the solid resulted in a low-cost lithium hydroxide suitable for vacuum thermal reduction. The yield of lithium hydroxide from the spodumene was about 80 per cent.

The lithium hydroxide was briquetted with ferrosilicon and lime in the proportions 1FeSi (80 per cent Si) :2.7LiOH: 4CaO and heated to 1100 C at a pressure of 1 to 25 microns. The reaction was probably as follows:

$$\mathrm{24LiOH \rightarrow 12Li_2O + 12H_2O}$$

$$\mathrm{12Li_2O + FeSi_6 + 15CaO}$$
$$\mathrm{\rightarrow 24Li + 3Ca_3SiO_5 \cdot 3Ca_2SiO_4 + Fe}$$

[redacted]

The lime was necessary to prevent the formation of lithium silicate, which reduced the yield to about 40 per cent. With lime the yield was 85 per cent to 90 per cent, or an overall yield of metal from the ore of about 70 per cent. Redistillation of the metal resulted in a product above 99 per cent purity. Based on a production of 2,000 pounds of lithium metal a day, it was estimated that the cost (without profit) would be about $3.00 a pound. The cost of the hydride would be little, if any, higher than that of the metal.

About the time this process was developed the demand for lithium for sea-rescue kits fell off, and the production became available for meteorological use. Even though the production cost by the new process was much less, materials for new plants could not be made available as long as the existing electrolytic plants could meet the demand.

7.4 SODIUM HYDRIDE-ALUMINUM MIXTURES[4,5,6,7,9]

An investigation of sodium hydride as a material for generating hydrogen disclosed that the pure material was unsatisfactory for field use because of its extreme reactivity. It is a fluffy powder which ignites spontaneously when exposed to air. Even when pelleted, its reaction with water was too violent to control. It was found that the addition of 25 per cent oil resulted in a product the reaction of which could be controlled, but the gas produced per pound dropped accordingly, and the oil was difficult to clean from the equipment and was also carried into the balloon by entrainment, necessitating the use of a filter.

The substitution of granular aluminum for the oil gave a product even more stable than the oil mixture, but which produced as much hydrogen per pound as the pure hydride. The aluminum had the further advantage of neutralizing the caustic soda formed, so that the water solution of the residue was not hazardous to handle. A mixture containing 50 per cent aluminum (200 to 300 mesh) gave the best results. Grade 3 secondary aluminum (93 to 94 per cent purity) was used satisfactorily.

Sodium hydride was produced by the hydrogenation of sodium according to the following equation:

$$2\text{Na} + \text{H}_2 \rightarrow 2\text{NaH} + 28{,}000 \text{ calories}$$

The sodium was hydrogenated in the presence of the aluminum powder which served as a dispersing agent for the sodium and resulted in an intimate mixture of the aluminum and sodium hydride in the final product. It was found that magnesium stearate increased the speed of hydrogenation and also acted as a binder for the pelleting. The amount of stearate required was approximately 0.12 per cent based on the final product. The reaction was carried out in a jacketed autoclave, equipped with an agitator, and connected to a Dowtherm circulating system for heating or cooling the contents. The sodium was hydrogenated with hydrogen at 10 to 15 pounds pressure and with the reactor contents maintained at 250 to 300 C.

After heating to the desired temperature, cooling was necessary to remove the heat of reaction and to maintain the temperature at this point. When the reaction was completed, only about 20 percent of the product was withdrawn; the remainder was left for dispersion of the new charge. The material discharged was cooled, pelleted, and packaged in an inert atmosphere to prevent reaction with oxygen and moisture in the air. A 30-gallon autoclave produced approximately 4½ pounds of product per hour.

Using secondary aluminum, the composition of the final product was approximately as listed below.

Sodium hydride	50%
Aluminum	46%
Inerts (chiefly metallic impurities present in the aluminum)	4%

This mixture reacted with water as follows:

$$\underset{(0.50\text{ lb})}{1.25\text{NaH}} + \underset{(0.46\text{ lb})}{1.00\text{Al}} + \underset{(0.04\text{ lb})}{\text{Inerts}} + \underset{(0.682\text{ lb})}{2.25\text{H}_2\text{O}}$$
$$\rightarrow \underset{(1.397\text{ lb})}{1.00\text{NaAl0}_2} + \underset{(16.63\text{ cu ft})}{2.75\text{H}_2} + \underset{(0.151\text{ lb})}{0.25\text{NaOH}} + \underset{(0.04\text{ lb})}{\text{Inerts}}$$

Thus 1 pound of mixture will theoretically produce 16.63 cu ft STP of hydrogen. Approximately 220 Btu is evolved for each cubic foot of hydrogen produced.

A generator was developed to meet the de-

[redacted]

mands of the Signal Corps for filling their meteorological balloons, using the sodium hydride-aluminum mixture. The balloons used had capacities of 6, 25, and 70 cu ft. The generator recommended (Figure 1) consisted of two parts: a cartridge (made in two sizes) containing the proper amount of sodium hydride-aluminum mixture to fill the 6- and 25-cu ft balloons, and a scrubber which cooled and washed the gas. The cartridges were designed to be discarded after use whereas the scrubber was designed for reuse with cartridges of either size.

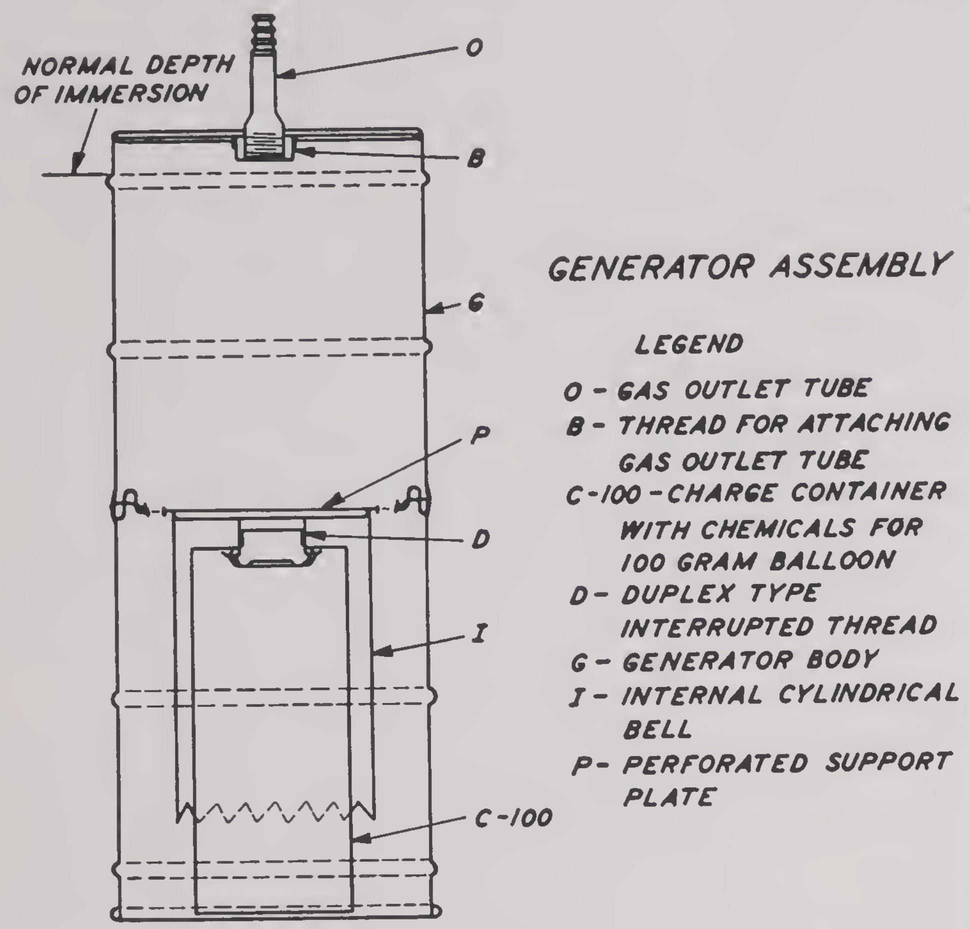

NORMAL DEPTH OF IMMERSION
O
B
G
P
D
I
C-100
GENERATOR ASSEMBLY
LEGEND
O - GAS OUTLET TUBE
B - THREAD FOR ATTACHING GAS OUTLET TUBE
C-100 - CHARGE CONTAINER WITH CHEMICALS FOR 100 GRAM BALLOON
D - DUPLEX TYPE INTERRUPTED THREAD
G - GENERATOR BODY
I - INTERNAL CYLINDRICAL BELL
P - PERFORATED SUPPORT PLATE

FIGURE 1. Generator assembly designed for the chemical generation of hydrogen to fill meteorological balloons.

The scrubber was cylindrical in shape and weighed about 2.8 pounds. It was 16 in. high and $6\frac{1}{2}$ in. in diameter. An internal cylindrical bell $4\frac{1}{4}$ in. in diameter was attached in the bottom half of the outer cylinder. The scrubber was open at the bottom and was closed at the top, except for a gas outlet tube. The 6-cu ft cartridge was $2\frac{1}{2}$ in. high by $3\frac{3}{8}$ in. in diameter and weighed 0.8 pound including 0.4 pound of charge. The 25-cu ft cartridge was $7\frac{1}{4}$ in. high by $3\frac{3}{8}$ in. in diameter and weighed 2.3 pounds, including 1.5 pounds of charge. For filling the cartridges the hydride aluminum was compressed into pellets $2\frac{7}{8}$ in. in diameter and $\frac{3}{4}$ to 1 in. thick.

In operation, a pull tab was removed from the bottom of the cartridge exposing a $\frac{1}{2}$-in. diameter hole, and a friction cover was removed from the top exposing a series of $\frac{17}{64}$-in. holes. The cartridge was then attached inside the cylindrical bell of the scrubber by means of a quarter-turn coupling. The generator assembly was immersed in water to within 1 in. of the top. Water entered the cartridge through the hole at the bottom and reacted with the chemical. The hydrogen generated was emitted from the cartridge through the holes at the top, was deflected downward by the internal cylindrical bell, and passed under the saw-toothed edge of the bell. The gas then flowed upward through the water in the space between the bell and the outer wall of the scrubber, disengaged from the water surface in the space above the bell, and passed through a hose to the balloon. In passing through the water the gas was washed and cooled. The small cart-

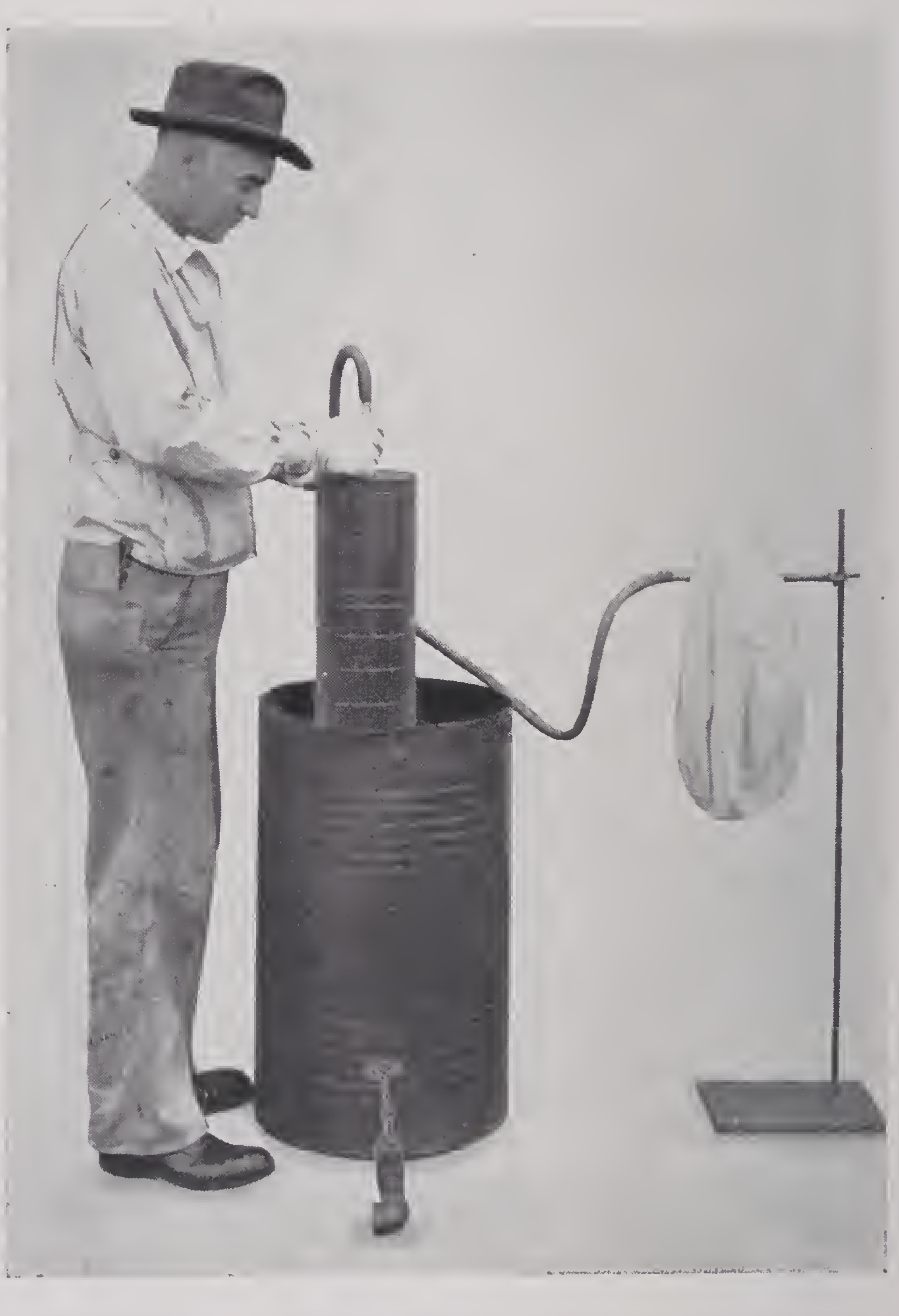


FIGURE 2. Operation of chemical hydrogen generator.

ridge produced hydrogen at the rate of approximately 1¼ cfm and the larger cartridge at the rate of approximately 2¼ cfm. Large balloons could be filled by discharging three large cartridges consecutively or in series.

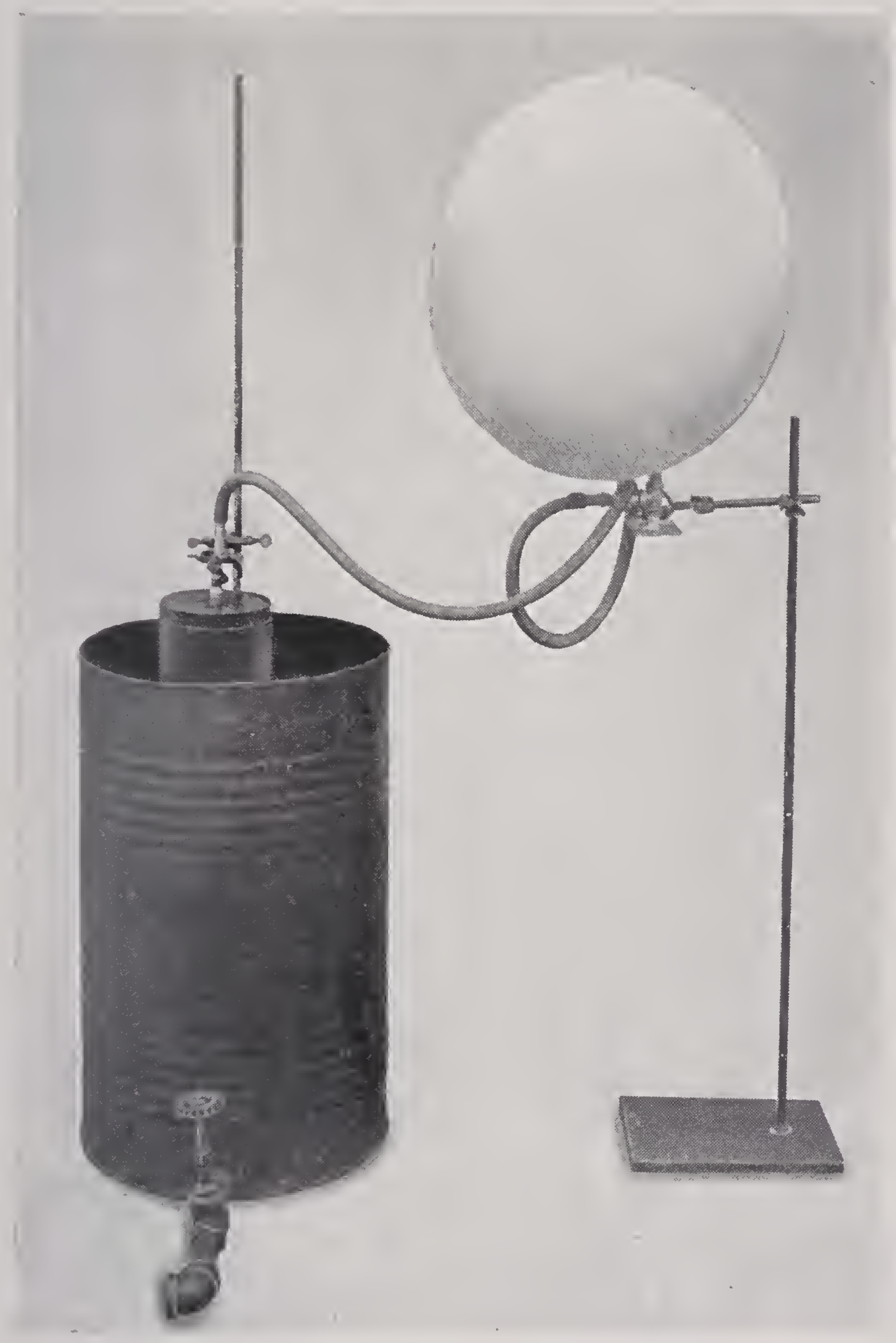


FIGURE 3. Operation of chemical hydrogen generator to inflate balloon.

Figures 2 and 3 illustrate the operation of the generator by immersion in water contained in an open drum.

The following table gives data on the rise in alkalinity and temperature of the water which was used for four consecutive generators of 25-cu ft charges of sodium hydride-aluminum mixture. The data are based on the use of 250 pounds of water, originally at 63 F.

Generator Number	Wt Chemical Used (Grams)	Final Water Temp F	Maximum Gas Temp F	Free NaOH
1	789	92	101	0.12%
2	782	120	126	0.24%
3	790	147	174	0.36%
4	790	165	189	0.48%

7.5 INVESTIGATION OF OTHER CHEMICALS[2]

Pure calcium hydride was produced for the British by hydrogenation of calcium metal. This material was satisfactory, but because electric furnaces were required in producing the metal, it was expensive and the supply limited.

A process was available for producing an impure calcium hydride by the reduction of lime with aluminum or magnesium according to the following reactions:

$$3CaO + 2Al + 3H_2 \rightarrow 3CaH_2 + Al_2O_3$$
$$CaO + Mg + H_2 \rightarrow CaH_2 + MgO$$

The hydride produced in this way is only about 40 per cent pure. However, it reacts with water satisfactorily to produce hydrogen.

Because of the large volume of hydrogen theoretically produced from a unit weight of sodium borohydride, an investigation was made of the practicability of producing it. Because of the high cost of lithium, the lithium borohydride was not considered feasible. Two possible methods for producing sodium borohydride were considered.

7.5.1 First Process

1. Preparation of sodium trimethoxy borohydride.
   a. $2Na + H_2 \rightarrow 2NaH$.
   b. $3CH_3OH + B_2O_3 \rightarrow (CH_3O)_3B + H_3BO_3$.
   c. $NaH + (CH_3O)_3B \rightarrow NaBH(OCH_3)_3$.
2. Preparation of diborane.
   a. $6NaBF_4 + B_2O_3 + 6H_2SO_4 \rightarrow 8BF_3 + 6NaHSO_4 + 3H_2O$.
   b. $BF_3 + (C_2H_5)_2O \rightarrow BF_3 \cdot (C_2H_5)_2O$.
   c. $6NaH + 8BF_3 \cdot (C_2H_5)_2O \rightarrow B_2H_6 + 8(C_2H_5)_2O + 6NaBF_4$.
3. Preparation of sodium borohydride.
   a. $2NaBH(OCH_3)_3 + B_2H_6 \rightarrow \underline{2NaBH_4} + 2(CH_3O)_3B$.

7.5.2 Second Process

1. Preparation of sodium trimethoxy borohydride, as above.
2. Preparation of sodium borohydride.

$$4NaBH(OCH_3)_3 \xrightarrow{heat} NaBH_4 + 3NaOCH_3 + 3(CH_3O)_3B.$$

[redacted]

In the second process the borohydride must be separated from the final mixture by dissolving it in pyridine or liquid ammonia, filtering and evaporating off the solvent.

A preliminary engineering study indicated that both processes were too complicated for the borohydride to compete with the sodium hydride-aluminum mixture on a cost basis. In each case the starting point was NaH as was used directly in the sodium hydride-aluminum mixture.

Further work by the Army Signal Corps disclosed that pure sodium borohydride was not sufficiently reactive with water for field generation of hydrogen. In order to correct this, it was necessary to add an acidic material, such as boric oxide, and this reduced the amount of hydrogen produced per pound of material. The borohydride has the advantage of being less pyrophoric than most of the others considered, and is therefore safer to handle in case of accidental breakage of the containers. Sodium hydride is poorest from this standpoint.

Chapter 8

PLANE CRASH DYE MARKER

8.1 SUMMARY

As an aid in sea rescue work a dye marker was developed which was ejected automatically as the airplane hit the sea. The ejection was accomplished by squibs actuated electrically through a switch which was closed by sea water. The floating container carried uranine dye compounded with polyvinyl alcohol to control and extend the time of solution of the dye in sea water. The mark produced was visible from 5 to 8 miles at 10,000 ft and persisted for 16 hours.

8.2 INTRODUCTION

Search parties looking for survivors of a plane crash at sea had a very difficult task unless there was some way of knowing the approximate location. A dye marker had been proven an effective aid for attracting planes, and was carried in life jackets and as part of the equipment in all life rafts. The dye used was powdered uranine, the soluble sodium salt of fluorescein. The dye was released by the survivor when a plane was sighted, producing a brilliant yellow fluorescent patch on the water. This mark lasted for ½ to 1 hour and if the plane did not see it, no more dye was available. Occasionally the survivors in the rafts were unconscious and unable to release the dye or use other available signal devices.

Because of this situation a project was undertaken for the development of a device which could be attached to a plane without impairing its performance, and which would be released automatically if the plane crashed in water, producing a mark visible for about 8 miles at an altitude of 10,000 ft and lasting a minimum of 16 hours. The weight of the unit had to be small enough so that it could be carried on any type of plane.

8.3 DYE COMPOSITION

Considerable previous work had indicated that uranine was as effective a marking material as could be found, but when used as an unconfined powder the mark was soon dissipated by motion of the water. It was decided, therefore, to use uranine rather than to search for a better material, but to devise some method of controlling the solution of the dye.

Experiments showed that from 0.3 to 0.4 pound of dye per hour, released continuously, was required for a mark having the desired visibility. Attempts were made to control the solution to the desired rate by enclosing the dye in a cloth bag, but the untreated dye still dissolved too fast. A coating of stearic acid on the dye retarded the rate of solution too much. All the rates obtained with this bag method, however, varied more with varying intensity of wave action than could be tolerated. Following these tests the bag method was discarded and the use of a solid cake of uranine decided upon.

A number of water-soluble binding agents were tried and several were satisfactory. The mixture recommended was:

Uranine (Calco 86% or equivalent)	87.5%
Polyvinyl alcohol (high viscosity)	1.0%
Water	11.5%

The polyvinyl alcohol was dissolved in the water and mixed with the uranine in a heated dough mixer until a uniform dough was obtained. The mixture was then pressed into the two open ends of a 4-in. aluminum tube having an air space in the center of the tube to give the cylinder buoyancy. Methyl cellulose and Cellosize were as effective as polyvinyl alcohol as binders. The inclusion of a binder was necessary to prevent a "honeycombing" effect as the dye dissolved. It was later determined that somewhat better results were obtained by mixing uranine with 3 or 4 per cent of water and 1 or 2 per cent of binding agent and pressing it under 6 to 8 tons per sq in.

Trials of this dye-filled cylinder indicated that the rate of solution was about right at the start, but that less dye was needed to replenish that which dissipated after the mark was established. The cylinder as finally designed (Figure 1) was 4 in. in diameter and 16 in. long. The dye compartments (one in each end) were in the shape of a frustum of a cone, 4 in. in diameter at the large end, $1\frac{1}{2}$ in. in diameter at the other end, and 8 in. long. The 4-in. ends were welded to the open ends of the cylinder, leaving an air chamber around the dye containers. As the dye dissolved, a continually reduced surface was presented to the water. Sea trials showed that this unit, which contained 6 pounds of dye mix, gave a satisfactory mark for 16 to 18 hours.

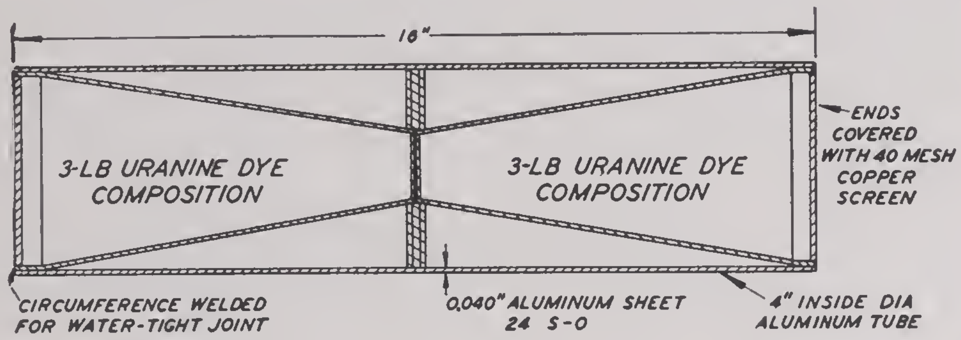

16"
3-LB URANINE DYE COMPOSITION
3-LB URANINE DYE COMPOSITION
ENDS COVERED WITH 40 MESH COPPER SCREEN
CIRCUMFERENCE WELDED FOR WATER-TIGHT JOINT
0.040" ALUMINUM SHEET 24 S-O
4" INSIDE DIA ALUMINUM TUBE

FIGURE 1. Dye-filled cylinder serving as sea rescue marker.

8.4 RELEASING MECHANISM

The releasing mechanism designed for automatic ejection of the dye was actuated by a water switch which was closed by sea water when submerged. Two dry-cell batteries detonated three electric squibs, which blew off the catches. Two springs ejected the dye container. The unit was designed to be mounted inside the plane's surface, with the panel through which the dye was ejected mounted flush with, and acting as part of, the skin of the plane. Suitable traps were placed between the water switch and the entrance for the water so as to avoid any possibility of premature operation due to rain or splashing of water. Trials showed that the release mechanism was activated within 7 sec of contact with water. The complete model weighed approximately 9 pounds.

A second unit was developed for mounting externally. On planes where a slight added air resistance could be tolerated, this unit had the advantages of simpler design, easier installation, and of being mountable on any type of plane without modification. The operating and releasing principles were the same as for the internally mounted model. The design consisted of a teardrop-shaped shell $17\frac{1}{8}$ in. long and $6\frac{1}{8}$ in. at its largest diameter. The forward section was sealed off as a buoyancy chamber and the rear section filled with 6 pounds of dye cake. Between these two sections was the releasing mechanism previously described. The shell was fastened to a base plate permanently attached to the plane. The squib released the shell from the base plate exposing an opening to the dye chamber.

The shape of the dye container of this model, as well as the location, shape, and size of the area of dye exposed to the water, differed markedly from that of the internally mounted design. This would in all probability require a different dye composition. No tests at sea were made with this unit and the necessary modifications in the dye composition were not worked out.

Chapter 9

DEVELOPMENT OF OXYGEN MASKS

9.1 SUMMARY

As a result of a study made in the fall of 1940, it was evident that the oxygen masks then in use were not usable at high altitudes and low temperatures. A new molded rubber mask was designed and tested, and shown to be satisfactory in all important respects. This mask represented a very great advance in its ability to operate at very low temperatures, and was adopted and procured in quantity as the Army A-9 mask immediately after Pearl Harbor.

A subsequent requirement of a full head coverage mask was met by a development involving an eye and an oro-nasal cavity with a check valve between, arranged so that the fresh air or oxygen was first drawn over the double plastic eyepieces. This mask operated satisfactorily without fogging of the eyepieces at −50 F.

9.2 INTRODUCTION

Before the start of World War II it was realized that the oxygen mask then in use was not satisfactory for extended periods at the high altitudes and low temperatures. A study of the problem indicated that the following points should be considered in constructing an oxygen mask:

1. Comfort.
2. Interference with vision.
3. Mask dead space.
4. Marginal leakage.
5. Effects of cold.
6. Arrangements for microphone.
7. Nonirritation of face in heat and cold.
8. Mask suspension—ability to loosen with one hand.
9. Resistance to breathing.
10. Characteristics and quality of mask rubber.
11. Location of oxygen ports and exhalation valve.
12. Oxygen supply regulator.

Before the development of a new mask was started, the efficiencies of the three military masks then in use were determined. The first of these masks was the MSA Type A mask (Navy White Long). This mask was long and narrow, made of rather inelastic rubber, and had a very narrow bearing surface upon the face. It required extremely tight strapping to be made even approximately tight. The mask was intended for use with the MSA self-contained oxygen apparatus. It contained no valves, and it was impossible to avoid leaks around the bridge of the nose and around the corners of the mouth, which were particularly prominent during talking. It was unsuitable for use with a closed-circuit oxygen apparatus and could not be used with any oxygen supply system where waste of oxygen was a matter of any moment. A leaky mask such as the Navy White Long can be employed only where oxygen is delivered to the mask under slight positive pressure and a constant degree of outward leakage is expected.

The second mask tested was MSA Type B mask (Navy White Short). This mask could be made quite tight if sufficient tension on the straps was used, but it was held to the face by the straps rather than being fitted to the face. Leakage was preventable, but leaks around the bridge of the nose frequently occurred. On testing in the low-pressure chamber, the mask was regarded as unsatisfactory, and would be very dangerous in the presence of severe cold since ice formation readily occurred inside the mask at subzero temperatures.

The third mask tested was the BLB oro-nasal mask Type A-8. This mask used a disk of sponge rubber as the exhalation valve, and inspiration was accomplished by taking oxygen from a rubber bag pendant from the lowest part of the mask, the oxygen being supplied continuously to the bag by an automatic reducing valve. The mask was fragile, and the breathing bag very light. It could be made to fit if strapped tightly, but was uncomfortable. If used under quiet circumstances at altitudes not above 17,000 ft in cabins warmed so as to prevent ice formation, the BLB oro-nasal mask was considered safe. In military aviation, where higher altitudes must be met and where subzero temperatures are regular, the mask proved unsuitable, the sponge-rubber exhalation valve freezing regularly and extensively so that practically no air could be forced through it.

For testing these masks and the masks later developed, it was necessary to work out suitable methods. The determination of points of mask leakage was made by means of the well-known coal-dust spray technique. Mask leakage was also tested by a helmet technique during normal breathing, during increased breathing due to inhalation of increased CO_2, and during continuous talking.[4]

In addition to the testing of the masks, the efficiency of the MSA self-contained oxygen apparatus was also determined. In this apparatus, which was for use with the MSA Type A mask, oxygen was generated chemically in a canister containing a compound which gave off oxygen. The carbon dioxide and water vapor were removed by the reaction which liberated oxygen. The apparatus was a closed-circuit device and was said to provide about 45 liters of oxygen for the small canisters supplied, and 65 liters for the larger canisters. In such an appliance the subject breathes through the canister, and it is essential that no serious development of resistance occur during use. The principal defect of the appliance was the development of resistance frequently greater than 10 cm of water while the subject was at rest, and intolerably high during mild work. Owing to changes in resistance and difficulty of access of water vapor to the chemical in the canister, the oxygen supply provided by it was neither constant nor, at times, adequate. These defects were so pronounced at sea level and under normal room temperature that its performance was not tested at low temperatures or under conditions of low barometric pressures.

9.3 DEVELOPMENT OF THE L-12 MASK

With this background concerning the important features of a satisfactory oxygen mask and the faults of the existing masks, a new mask, designated the L-12, was designed. Figure 1 is an illustration of this mask. In order to prevent leakage of the mask, there was a large surface in contact with the face and the mask was carefully shaped around the nose and chin so that a tight fit was obtained without the necessity of tight strapping. There is no region where masks become more uncomfortable than across the nasal bridge, if hard pulling is required to prevent leakage. A loop of wire is necessary for most wearers in order to assure absolute tightness about the nose. The ports for entrance of oxygen or oxygen mixtures were at a distance from the exhalation valve, so that the cold entering oxygen would not freeze the water vapor in the exhaled air, with consequent plugging of the valve and obstruction to breathing. For the same reason, the exhalation valve was placed as near the mouth as possible in order to take advantage of the maximum warmth of the expired air for the prevention of ice formation. In the presence of wind a shield was necessary to protect the expiratory valve to avoid ice

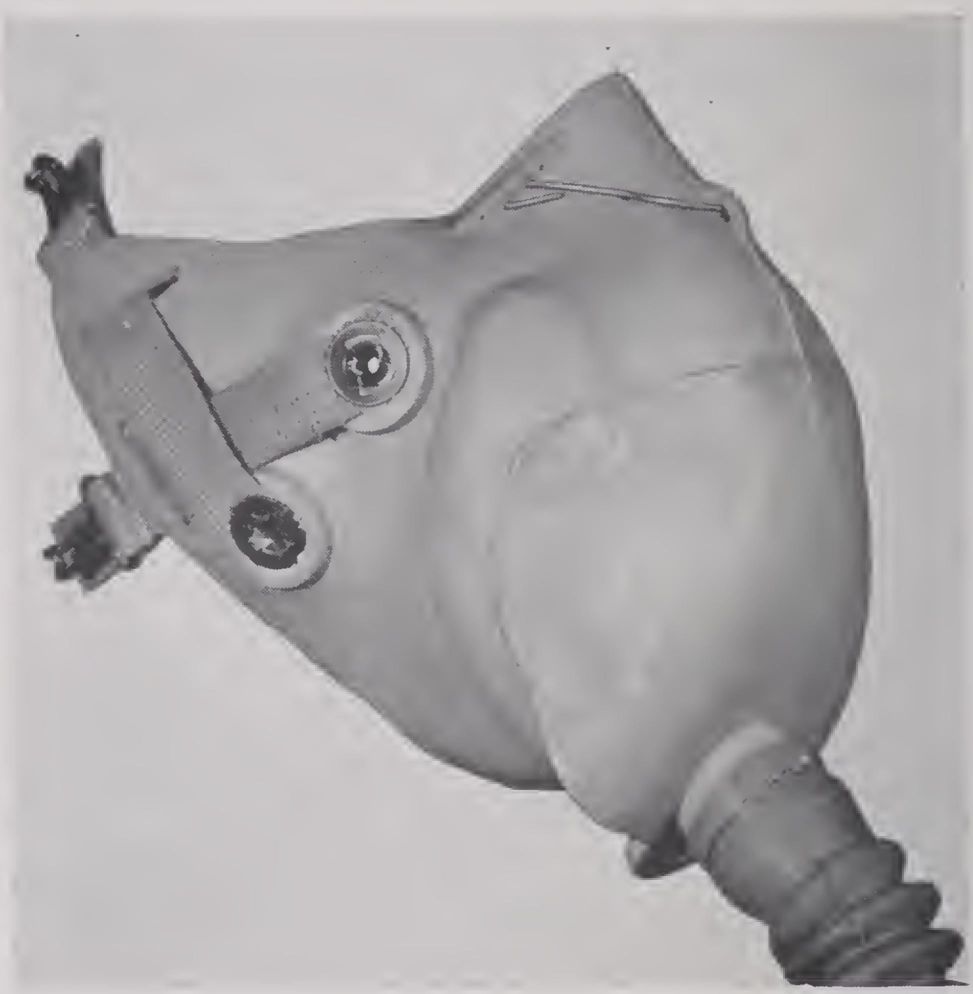


FIGURE 1. Three-quarter view of fully equipped L-12 oxygen mask.

formation in the exhaled air. This shield was molded from the same type of rubber used in the face piece, and was cemented on, being so formed as to fit exactly. There were no surface obstructions which interfered with vision, and there was no difficulty in wearing the Army goggles with the mask. The rubber used had had a long period of testing in Army gas masks and was known not to cause irritation or allergy. The dead space in the mask varies for different facial contours, but it rarely is above 100 cu cm.

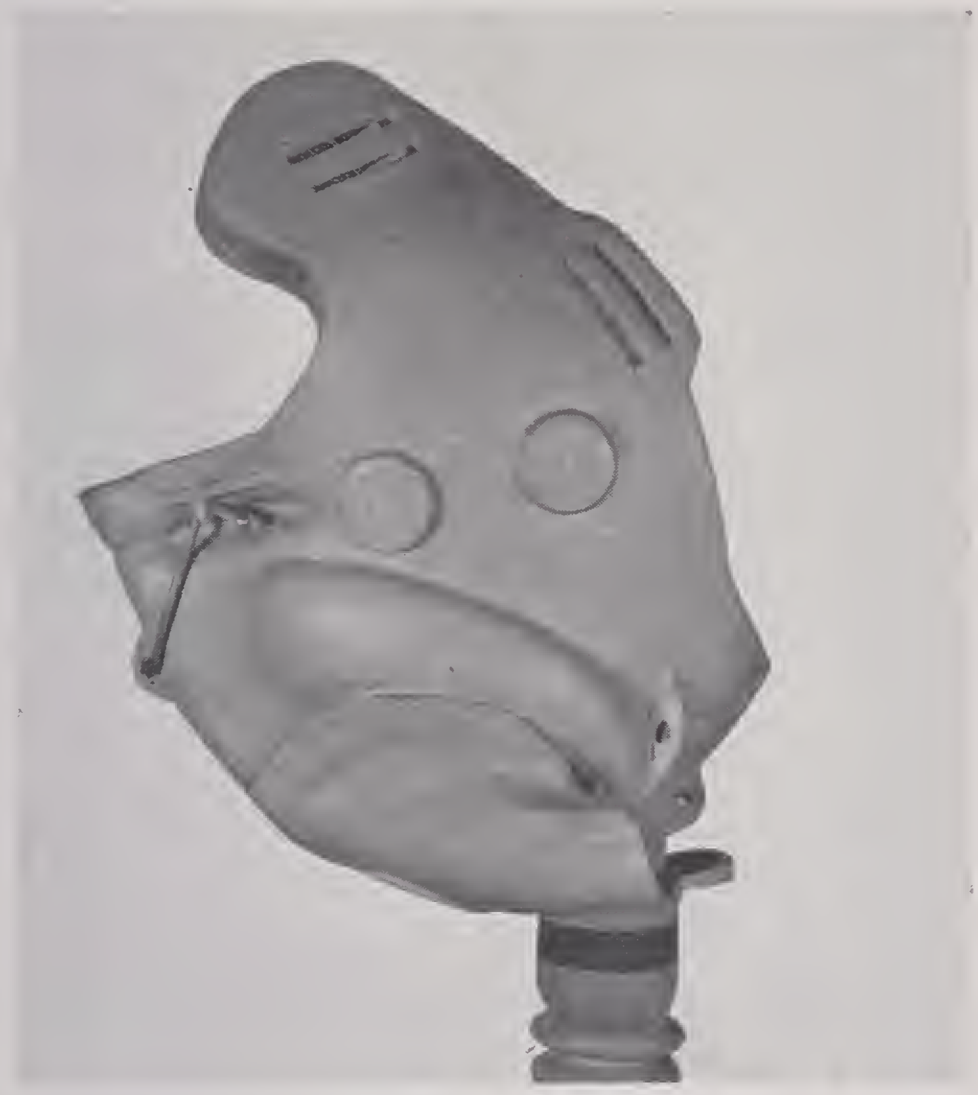


FIGURE 2. Side view of A-9 oxygen mask without straps.

Leakage tests on the L-12 mask showed it to be satisfactory from this standpoint, and tests in the low-pressure chamber indicated that subjects at rest and light work received adequate oxygen through the mask at altitudes up to 40,000 ft and experienced no discomfort from the mask. The mask was outstanding in its ability to withstand extremely low temperatures for several hours.

Field and laboratory work on this mask sponsored by the NDRC and the Army Air Forces resulted in changes in the suspension, in the method of securing a tight fit about the nose, and in the construction of the expiratory valve and oxygen inlet tube. After these changes had been incorporated, the mask was adopted by the Army as the A-9, and commercial production was begun in January 1942. This final mask is shown in Figure 2. The Army Air Forces later made modifications of the A-9 mask which resulted in the A-10 mask. Similar masks were later developed by the Mine Safety Appliance Company for the Navy and by the Canadians for the RCAF.

9.4 THE FULL-FACE OXYGEN MASK

These masks covered only the lower part of the face, and, when worn with goggles, left part of the face exposed. Occasionally freezing resulted from extended exposure to very low temperatures. To prevent this, a full-face oxygen mask was developed, with fogproof goggles incorporated so that the vision would be unobstructed by fog or frost at subzero temperatures. The principles upon which this development was based may be listed as follows.

1. Separation of the mask into two compartments: one, the eye compartment, and the other the oro-nasal compartment.
2. Controlled one-way ventilation of the eye compartment by the inhaled air of the wearer for the removal of the water vapor formed by the eyes and surrounding skin, which vapor would, by saturating the air over the eyes, cause lens fog.
3. Exclusion of the saturated exhaled air from the eye compartment where the added water vapor arising from the lungs would intensify the formation of lens fog, the exclusion being accomplished by a valved connection between the eye and oro-nasal compartments as well as by a nasal sealing ridge built into the mask between the two compartments.
4. The use of a double plastic lens, embodying an air cell 0.060 in. thick, by means of which the outer and inner surfaces of the lenses are effectively insulated one from the other, thus greatly reducing the loss of heat from the eye compartment.
5. The use of an absorbent (antifog) coating on the surface of the lens nearest the eye to prevent the formation of intermittent fog

during the exhalation period when at temperatures lower than 30 F.

6. The elimination of external clamps used for sealing the lenses in the mask, by making use of the elasticity of the rubber as well as building into the eyepieces flexible metal beads to prevent the accidental removal of the lenses.

7. The shielding of the exhalation valve after the manner of the A-10 series of oxygen masks to prevent freezing of the expiratory valve, except that the shield was integrally molded with the face piece instead of being cemented.

Without the aid of externally applied heat, the limit of usefulness of the mask was around −50 F, though tests at Wright Field gave fairly satisfactory results at a temperature of −91 F. The application of external heat by means of an electrical heater in the oxygen inlet tube theoretically allowed satisfactory use of the mask to any desired subzero temperature.

The lenses of the final mask were $2\frac{10}{16}$ in. in diameter. Perimeter tests on the visual fields, conducted by the Aeromedical Laboratory, Wright Field, showed this mask to be better than the B-8 goggles.

After a few changes proven necessary by field tests, three sizes of the mask were constructed and were standardized by the Army as the A-16. This mask is shown in Figure 3.

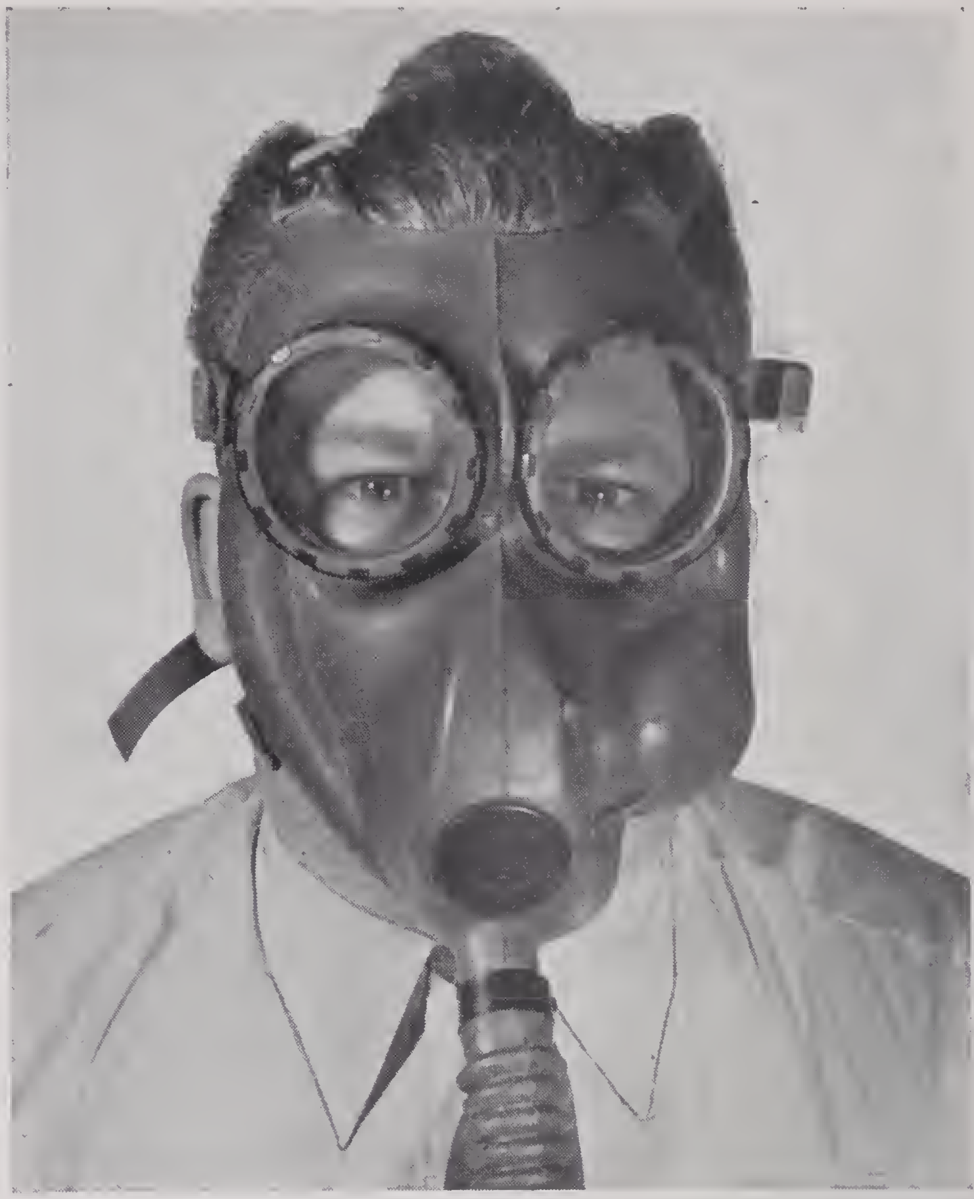


FIGURE 3. Front view of A-16 full-face oxygen mask showing large eyepieces, eye clamps, and the method of molding valve-shield as an integral part of the mask.

Chapter 10

LEVINSTEIN-H

10.1 SUMMARY

Mustard gas produced by the standard Levinstein process is impure, relatively unstable, and has physical properties which are undesirable for many tactical uses. For these reasons methods were developed by which the Levinstein product (H) might be purified. Additives were developed to reduce the shatter of mustard when used as an aircraft vesicant spray and to produce a rain of controlled drop size. A great many data were obtained on the stability of the Levinstein product and of the thickened agent. Data on evaporation of drops and drag coefficients for falling drops were employed to determine the loss due to evaporation from aircraft spray. A new mixed agent involving the addition of small quantities of white phosphorus to mustard was partially developed. This showed remarkable promise for the rapid production of concentrations of mustard vapor high enough to be lethal to masked troops in a very short time.

Laboratory and pilot plant studies showed the feasibility of purifying H by pentane distillation, fractional melting, pentane extraction, pentane detarring, steam distillation, or vacuum distillation. Table 1 indicates the pressure stability of several of the products.

Table 1. Gauge pressures (psi) developed during storage at 65 C in contact with iron.

	Number of days at 65 C		
	20	40	60
Crude Levinsteins (average of 18)	17	. .	. .
Steam distilled (brick semi-works)	11	17	20
Crude Levinstein plus 1 per cent hexamine	7	12	20
Thiodiglycol H, plus 1 per cent hexamine	3	6	9
Pentane detarred, plus 0.2 per cent hexamine	2	5	. .
Steam distilled (laboratory glass column) plus 1 per cent hexamine	1.5	2	3
Steam distilled (ceramic column), no inhibitor	. .	2	2.5

Plant design estimates indicated steam distillation to be the simplest and cheapest process, and a joint report[21] by NDRC and the Chemical Warfare Service [CWS] Development Laboratory (September 16, 1943) recommended the installation of purification plants using steam distillation. The subsequent development that unused Lewisite plants might be converted for H purification led to the installation of a vacuum distillation unit.

Early work by the British showed that aircraft spray was most effective in producing casualties if the drops formed were in the range 1.5 to 5.0 mm diameter, and that the addition of chlorinated rubber and benzene to H led to the production of drops in this size range. In cooperation with CWS, groups working under NDRC made a search of possible thickening agents and developed test techniques to supplement the field trials. The thickened H mixture developed and adopted contained 0.6 to 0.9 per cent polymethyl methacrylate (scrap Lucite or Plexiglas), 1.7 per cent crude nitrogen bases, 12.7 to 12.4 per cent benzene, and 85 per cent H stabilized with 1 per cent hexamethylene tetramine. This mixture gave the desired drop size distribution, due to the structure rather than the viscosity of the liquid.

In connection with the several problems related to H, it was necessary to carry out numerous stability tests of H and H compositions under various conditions. The thickened H mixture described above is stable in lined M47 bombs for 3 to 6 months at 65 C (149 F).

In order to determine the amount of H which might evaporate in the use of high-altitude spray, data were collected on the evaporation of liquid drops and correlations obtained covering a wide range of conditions. The estimated evaporation of H in falling from 10,000 ft at 20 C is estimated to be 1 per cent for 6-mm drops, 8 per cent for 2-mm drops, 28 per cent for 1-mm drops, and 100 per cent for 0.5-mm drops.

It was found that the effective volatility of H might be greatly increased by incorporating 2 to 4 per cent of white phosphorus. This oxidized on contact with air and the temperature of the liquid H was raised more than 100 C. The result was copious evolution of a dense H smoke. Best results were obtained by the use of 15 per cent cyclic ethylene trithiocarbonate as a cosolvent, with the liquid held in the pores of small cubes of cellulose sponge. The development was not complete, as the sponge was not chemically stable in H, and the retention of the liquid by the sponges was rather low. The results indicated, however, that concentration-time dosages lethal to masked troops might be obtained in a very few minutes under conditions such that hours would be required with untreated H.

10.2 INTRODUCTION

Mustard gas, a powerful vesicant, was introduced as a chemical warfare agent by the Germans in 1917. In the early stages of World War II it became evident that this material would probably be the most important and widely used chemical agent if chemical warfare should break out, and various important problems concerning its tactical use were presented. The U. S. production was based on the Levinstein process and the product H was an impure material with an undesirably high freezing point, about 50 F, containing 70 to 75 per cent of the active agent, *bis*-β-chloroethyl sulfide. The large amount of impurities not only represented inactive diluent but resulted in poor storage stability at elevated temperatures, and presented serious obstacles to the development of new weapons and new formulations involving H. It is to be hoped that if H continues to be regarded as an important agent, the Levinstein process will be replaced by one of the alternative production methods by which pure H can be readily produced.

10.3 PURIFICATION OF LEVINSTEIN-H

10.3.1 Introduction

The importance of the stability of H on tropical storage and the necessity of developing a thickening agent for the anticipated use of high-altitude vesicant spray led to consideration in late 1942 of ways and means of obtaining pure mustard. Since U.S. production of H was based on the Levinstein process, it seemed more expeditious to develop methods of purifying the Levinstein product than to construct new plants for the manufacture of the pure agent. Accordingly, NDRC and CWS undertook simultaneously to develop and evaluate several processes for the separation of the pure mustard from the Levinstein product, of which large quantities were on hand.

10.3.2 Pentane Distillation

Because of the erroneous belief that hydrolysis of the H would be excessive if steam were employed, a study of vapor distillation using pentane vapor was initiated. The first equipment was of steel, and although coking was encountered in the packed towers, successful runs were made giving a product containing 97 per cent mustard on a pentane-free basis.[27] The steel equipment was redesigned with acid-proof brick lining in the towers and stainless steel piping, but the project was dropped before additional data were obtained because of the success of parallel studies of purification by other methods.

10.3.3 Fractional Melting

Small-scale experiments indicated the considerable promise of a novel purification method involving fractional melting. Solid H was partially melted and the two phases separated on a chilled vacuum filter. Although the efficiency of separation in a single step was not high, it seemed entirely practical to operate a multistage countercurrent system which would give high purity and good yield. Because of the success of the other processes, this method[22] was never carried to the pilot plant stage.

10.3.4 Steam Distillation

This method showed promise when tried on a laboratory scale in World War I, but was not developed at that time. Work by the CWS Development Laboratory in 1942 indicated the possible advantages of steam distillation, and both NDRC and CWS proceeded with the development of the process on a pilot plant scale. The first tests with stainless steel columns gave difficulties due to coke and tarry deposits; ceramic-lined packed towers were then substituted. As finally developed, the process[28] involved a short-time countercurrent contact of the crude H with superheated steam. The overhead vapors were condensed in a silver condenser, the water layer was discarded, and the mustard layer was dried, inhibited with hexamethylene tetramine, and stored in steel drums. The residue from the bottom of the column was discarded. No difficulty was encountered in operating the steam-distillation column for extended periods. Thus in a run for 146 hours with crude H from several lots, 57 pounds of 98 per cent mustard were obtained per 100 pounds of crude H containing 73 per cent mustard, which is equivalent to the recovery of 76 per cent of the *bis*-β-chloroethyl sulfide in the crude. Table 2 summarizes the operating data from two periods of this 146-hour run.[28]

Table 2. Operating data from steam distillation of crude Levinstein mustard. Six-inch ceramic column packed with 8.7 ft of $\frac{5}{8}$-in. Raschig rings.

Period	A	B
Number of hours	47	53
Feed rate, pounds crude H per hr	25.1	24.7
Steam rate, pounds per hr.	80	78
Steam temperatures, F	438	420
Bottom of packing, F	287	296
Distillate temperature, F	79	84
Distillate, per 100-lb feed	56.8	60.5
Residue, per 100-lb feed	39.3	37.3
Unaccounted, per 100-lb feed	(3.9)	2.2
Per cent H in feed (by vacuum distillation analysis)	77.5	71.4
Melting point of distillate, C	13.2	13.84
Purity of distillate, per cent H	96.6	98.4
Per cent recovery *bis*-β-chloroethyl sulfide	70.8	83.5

Similar results were obtained by CWS[33] with a semi-works tower of larger cross section, 13.5 by 13.5 in., which reduced heat losses and hence decreased steam required per unit of feed and product.

Table 3 is an abstract of the data, and it is evident that similar results were obtained in the two plants: a product purity ranging from 97 to 99 per cent mustard, containing 72 to 82 per cent of that in the crude H. The purity of the product appears to depend on variations in the nature of the low-volatility impurities rather than on the per cent mustard in the feed.

Table 3. Performance of steam-distillation pilot plants.

	Hours on test	Per cent mustard in crude H	Lb distillate per 100 lb crude H	Distillate purity, mol per cent	Per cent recovery
NDRC ceramic pilot plant					
Best feed	13.0	70.9	57.5	99.0	80.3
Worst feed	54.5	77.5	57.9	96.6	72.2
Average on test	146.5	72.9	57.2	97.9	76.8
CWS development laboratory, brick semi-works					
Best result	10.0	73.3	60.8	98.2	81.5
Average	42.5	74.0	53.8	96.5	70.2
Average	36.3	74.6	55.9	97.8	73.2
Average	34.0	73.4	55.5	97.4	73.7
Average on test	112.8	74.0	55.0	97.2	72.2

Edgewood stability tests[43] later showed that the distillate described in Table 2, when stored in uncoated M74 bombs at 65 C, does not corrode the bombs excessively nor is a significant portion of the steam-distilled H decomposed in six months' time.

10.3.5 Pentane Detarring

In this process the crude Levinstein-H is contacted with liquid pentane in a counterflow extraction tower at ordinary temperatures;

upon distilling off and recovering the volatile pentane from the purified mustard and inhibiting with hexamine, a stable product (see Table 1) is obtained.[29] This process gives a higher per cent recovery of the pure agent from the crude than steam distillation (96 per cent versus 75 per cent), but the product has lower purity (89 per cent versus 98 per cent). Both products inhibited with hexamethylene tetramine are very stable, and both processes were carried through the pilot plant stage. While both steam distillation and pentane detarring were successful, the advantage of the higher purity obtained by steam distillation was thought to be more important than the higher recovery obtained in pentane detarring.

10.3.6 Pentane Extraction

Because of the different chemical nature of mustard and the impurities contained in H, it appeared practical to separate the two by solvent extraction. An extraction column was operated in the laboratory using pentane as solvent, with a contacting section consisting of 6 ft of ¼-in. glass rings with feed near the middle and a "reflux" of depentanized product. Products containing 95 to 96 per cent mustard were obtained, but the calculated tower heights for plant-scale operation were very large. Extracts obtained, even with added hexamethylene tetramine, were less stable and more corrosive to iron than the products obtained by either pentane detarring or steam distillation.

10.3.7 Vacuum Distillation

Distillation at low temperature has long been the classical method of obtaining pure *bis*-β-chloroethyl sulfide in the laboratory, but it was not considered a practical plant process because of the complexity of the required vacuum equipment in relation to the simplicity of the steam distillation equipment. The availability of idle Lewisite plants made it possible, however, to proceed with large-scale purification of H by vacuum distillation of water-washed crude,[36,49,57] and a plant of this type was installed at Rocky Mountain Arsenal.

10.4 THICKENING OF H

10.4.1 Introduction

The technique of spraying a liquid from aircraft had been developed by the CWS in the years preceding World War II, and trials in Algiers and at Muroc Lake[1] had indicated that vesicant spray from high altitudes might be practical. Levinstein-H is relatively nonvolatile, but only the larger drops will reach the ground from high altitudes without being largely or wholly evaporated. In 1941 it was learned that the British had been able to increase the mean drop size of the spray by thickening the liquid H. This was accomplished by the addition of 2 per cent crepe rubber, or 8 to 10 per cent of chlorinated rubber, to H containing benzene, and the conclusion was reached that the amount of thickener should be such as to result in a viscosity of the thickened H of 6 poises at 10 C. It was urgently desired to develop a practical method of thickening H, and other vesicants, so as to obtain casualty producing spray from high altitudes. It was anticipated that such a thickened vesicant might also prove to be preferable for low-altitude spray, and that thickened agents might be employed in bombs and shells to increase the casualty producing effect of the dispersed liquid. Work on this problem proceeded with close cooperation between CWS and NDRC.

10.4.2 Selection of Thickening Agent

The use of rubber or its derivatives was ruled out because they were not available, and a large number of alternatives were studied by screening tests. As a group, the various synthetic polymers showed the most promise, but the majority proved to be insoluble in H. Others were soluble but caused little viscosity increase.[a] The most promising polymers appeared to be methyl methacrylate and polystyrene of proper types.

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[a] Among the 70 or more rejected materials were: polyvinyl chloride, chlorovinyl chloride, Neoprene, Vistanex, Polybutene B-12, Alvar, Formvar, Butvar, butyral vinylate, copolymer of vinyl chloride and vinyl acetate, butyl-isobutyl methacrylate, ethyl methacrylate, propylene glycol, diglycol stearate, Glucarine, Napalm, lignin, polystyrene (of low molecular weight), Venice turpentine, glycerin, and an equimolar mixture of 2-isovaleryl-1,3-indanedione and *n*-monodecylamine.

Figure 1 shows the viscosity of H containing varying percentages of several of the better polymers. (The unthickened material has a viscosity of about 0.08 stoke at 10 C.) Since the thickened solutions show non-Newtonian behavior, the viscosities reported vary with the rate of shear obtained in the test viscometer. It is evident that the polymer NDR 163 is effective in producing a viscosity of 6 poises at 10 C with only 1.1 per cent concentration by weight in H.

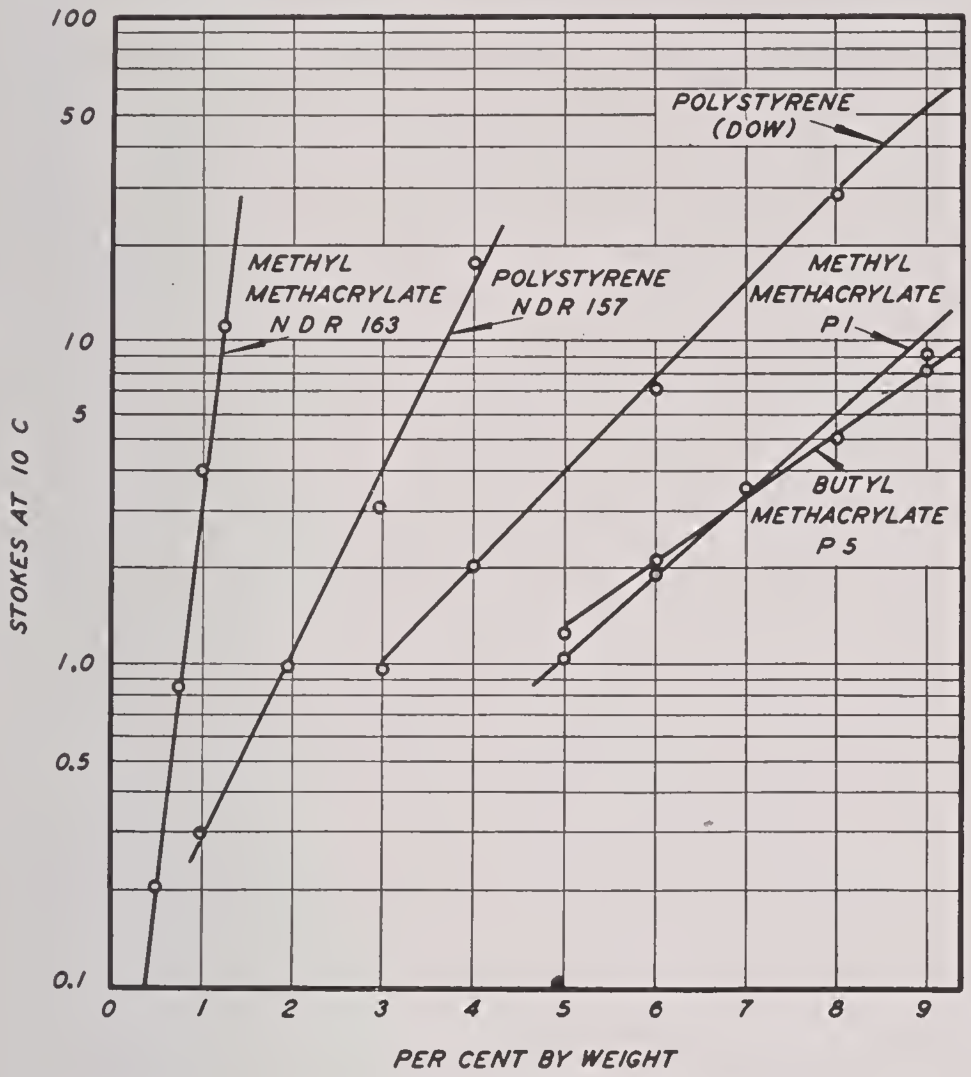

FIGURE 1. Viscosity of H containing several polymers.

Tests with polystyrene and with methyl methacrylate from various sources showed widely varying thickening powers, and this was traced to variations in molecular weight. The polymer P-1 is a commercial methyl methacrylate having a molecular weight of about 40,000, while the sample NDR 163 is an experimental polymer with a molecular weight of over 300,000. The greater thickening power of NDR 163 is evident from Figure 1. The same effect is noted in the case of polystyrene, where NDR 157 has the higher molecular weight.

The thickening effect as indicated by the viscosity was related to practical drop-size distribution in two ways: by a laboratory test mortar and by field trials with low-altitude aircraft sprays. The former[13,31] consisted of a small cylindrical cavity from which about 2 cu cm of sample was expelled by means of a piston driven forward by the explosion of a blank cartridge. The liquid was expelled horizontally and the drops formed collected on a test paper about 3 ft wide and 25 ft long. A count of the drop stain-size distribution and a correlation between drop and stain sizes yielded a drop-size distribution for the test material. Results with aircraft spray and with the mortar showed a reasonably good correlation between the results by the two methods, and the mortar was used in many screening tests to reduce the number of necessary field trials. Tests with the mortar showed that the percentage of the charge falling in the desired range of 1.5- to 5.0-mm diameter passed through a maximum as the molecular weight increased. In the case of methyl methacrylate this maximum is in the vicinity of 200,000. When methyl methacrylate sheet scrap (Lucite and Plexiglas) became available from aircraft factories, it was found to have a molecular weight varying from 100,000 to 380,000, and to be entirely acceptable as a thickening material. Methacrylate sheet scrap was adopted and standardized as the thickening material[50] to be used with H.

A number of the thickening agents showing the greatest promise on the basis of laboratory mortar tests and viscosity data were tested in a series of field trials at Edgewood in August 1943, using M-10 wing tanks at an altitude of 75 to 100 ft and an airplane speed of 200 mph. Drop cards were used to obtain drop stains and drop counts, and drop-size distributions were reported.[26] (See Figure 2.) One of the most significant results of these tests was the demonstration that viscosity alone was of little value as a criterion of proper thickening. It seemed clear that the degree of shatter of the liquid leaving the tail pipe of the spray tank[b] was dependent on the structure of the liquid imparted by the polymer, and that the quality of structure was more important than viscosity. Both British and NDRC workers[6,18,52] have attempted to isolate the rheological factors involved, but with only partial success. The field tests pointed to high molecular weight methyl methacrylate as the most promising thickener, but the effective concentration was shown to be approximately 0.75 per cent in H containing 15 per cent benzene, and this solution had a viscosity of only 0.5 poise—much less than the 6 poises originally thought necessary.

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[b] Shatter occurs and drop-size distribution is determined at this point; subsequent breakup of drops in falling through the air is negligible.

In the course of the exploratory work on possible thickening agents it was found that low concentrations of suspended fibers might be effective, and the mortar tests showed good drop-size control in the case of some samples of ground newspaper and asbestos fibers. After considerable laboratory study these were included in the 1943 field test program, but the results were disappointing. In the case of the fibers tested, the fiber concentration necessary to obtain good drop-size control was somewhat higher than the maximum for smooth discharge of the thickened material through the tail pipe of the spray tank. In view of the successful results with polymers, the experiments with suspended fibers were discontinued.

10.4.3 Preparation of Thickened H

When stored in munitions, thickened H [HV] is somewhat less stable than H without additive, and plant filling with HV seemed impractical where munitions might be expected to be stored in the tropics for a year or more. Accordingly, it was planned to adopt the procedure of "field filling" in which a solution of the polymer in benzene was added to a container of H in the field and incorporated by simple stirring. It was anticipated that this technique would be employed for filling aircraft spray tanks, and possibly M47 bombs. In preparing batches of HV for field trials at Dugway, it was found that certain plant batches of H were incompatible with the solutions of methyl methacrylate in benzene, and that a tarry deposit was formed. A large number of samples of stored H were obtained and tested, showing that this behavior was relatively common, especially in batches of H containing large amounts of iron. Most of the stored H showed more than the maximum of 0.25 per cent iron as $FeCl_2$ permitted by H specifications. Laboratory tests showed the formation of an insoluble iron-polymer complex, and it appeared that it might be necessary to remove the iron from the H to be thickened if methyl methacrylate were to be used. This could be done at the plant by ammonia treatment and by other methods,[56] but large stores of H existed which could not be plant processed.

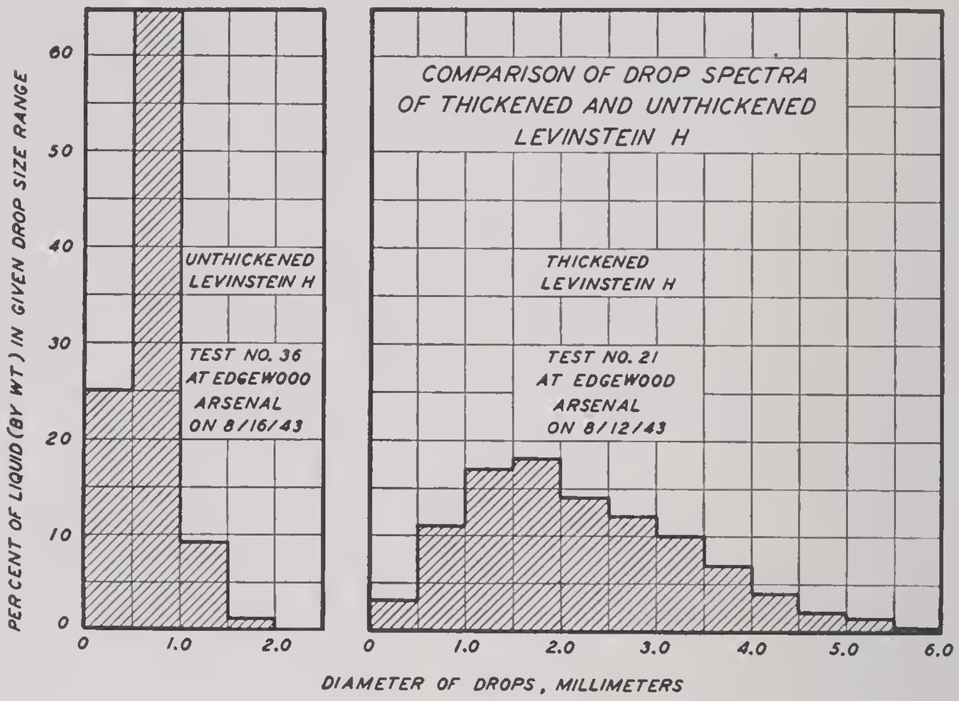

FIGURE 2. Comparison of drop spectra of thickened and unthickened Levinstein-H.

Fortunately, the problem of thickening high iron content H was solved by the discovery that certain pyridine-type nitrogen bases available in quantity as by-products from the petroleum and coal tar industries, served as effective iron deactivators and permitted thickening of the poorer grades of H with methyl methacrylate without deposition of a tar.

The specifications relating to the procurement of the polymer[38,50] called for a methyl methacrylate of such a degree of polymerization that a 5 per cent solution in benzene would have a viscosity of 8 to 25 stokes at 25 C. The VV solution is made[42] by dissolving in benzene 4 to 6 per cent of this polymer, ground to pass a 16-mesh screen, together with 11.3 per cent of crude nitrogen bases. The final HV is prepared by blending 15 parts by weight of the

VV solution with 85 parts of H containing 1 per cent hexamethylene tetramine (hexamine). Thus the final HV contains 0.6 to 0.9 per cent polymer, 1.7 per cent nitrogen bases, 12.7 to 12.4 per cent benzene, and 85 per cent of H stabilized with 1 per cent of hexamine. It has a viscosity of 0.4 to 0.6 poise at 10 C.

In order to avoid the use of a solvent such as benzene, it is possible to prepare a concentrated solution of polymer in distilled H [HD] and to use this in place of VV. This procedure, developed at Edgewood Arsenal, involves the preparation of a thick solution known as HVV, containing 2 per cent polymer and 10.0 per cent nitrogen bases. As in the case of VV, 15 parts of HVV are blended with 85 parts H, to produce an HV having a viscosity of about 0.5 poise at 10 C.

10.5 STABILITY OF THICKENED H[54,55]

Because of the inherent instability of H, the problem of storage was at all times one of great urgency and importance. CWS discovered the important effect of hexamethylene tetramine as a stabilizer, and essentially all stored H was stabilized with 1 per cent added hexamine. This inhibitor has a marked effect in retarding H decomposition, and is especially valuable in reducing or eliminating pressure development. The CWS agent HN-3 was also found to be effective in a similar way.

Since H is frequently acidic, and since iron salts accelerate H decomposition, the stability is much better in glass or lined containers than in steel. Suitable shell lining materials were developed under an NDRC contract (see Chapter 17) and specifications[24] issued. As might be expected, the stability of H in steel containers varies widely with the container size, the rate of decomposition varying roughly with the ratio of wetted surface to liquid volume. Stability in unlined 1-ton containers was found to be quite good, whereas the storage life of H in an unlined 75-mm shell at 65 C is less than one month. For these reasons the tests in 75-mm shell were considered valuable as representing the worst storage conditions.

The temperature 65 C was arbitrarily chosen for most stability tests, and a material standing up well for three months in 75-mm shell at 65 C was generally accepted as having satisfactory stability. Decomposition usually proceeds rapidly once it starts, so that curves of melting point and H content versus time are horizontal during an initial period and then dip sharply. For this reason stability results for several samples should not be compared on the basis of per cent decomposition after a definite time; a more significant comparison is that of the times to reach a specified per cent decomposition.

Levinstein-H containing 1 per cent hexamethylene tetramine is stable for four months at 50 C and for 6 to 8 weeks at 65 C in unlined 75-mm shell. It is probably stable for at least one year in unlined 1-ton containers in the tropics. The stability at 65 C in unlined 75-mm shell is increased to four months if hexamethylene tetramine is added directly to the shell. Ammonia-treated H is stable for three months at 65 C in unlined 75-mm shell without hexamethylene tetramine. The marked advantage of the lined shell is indicated by the fact that H containing 1 per cent hexamethylene tetramine lost less than 2 per cent in H content in six months at 65 C when stored in 75-mm shell lined with a bakelite-type lacquer. It is evident from these data that Levinstein-H has quite satisfactory stability if hexamethylene tetramine is used as an inhibitor and properly lined containers are employed.

The stability of pure mustard obtained by the thiodiglycol process or by the purification of H is much better than that of H. Thus thiodiglycol mustard shows only about 3 per cent decomposition in unlined 75-mm shell at 65 C in six months, but the pressures developed are similar to those obtained with H (200 to 300 psi). Hexamethylene tetramine reduces both decomposition rate and pressure development. The stability of steam- or vacuum-distilled H [HD] is similar to that of thiodiglycol mustard; the rate of decomposition is somewhat greater but the pressures developed are less. With only 0.2 per cent hexamethylene tetramine in steam-distilled HD, the loss in mustard content is but 0.4 per cent per month at 65 C, but the HD must be essentially water-

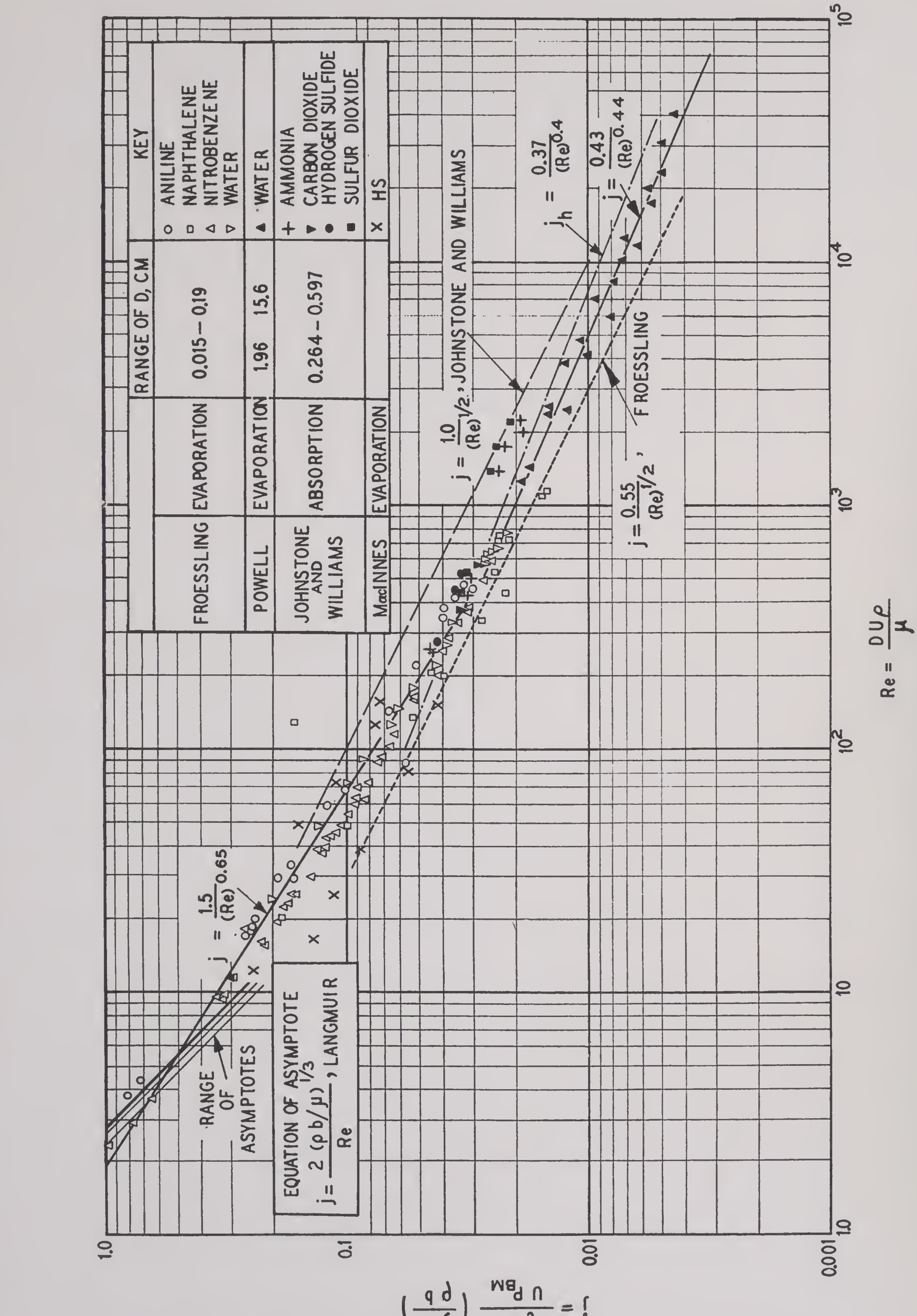

FIGURE 3. Correlation of data on diffusion to and from spheres.

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free if the hexamethylene tetramine is to be effective.

The stability of thickened H in contact with bare steel is poor at elevated temperatures. Laboratory tests have shown HV containing hexamethylene tetramine to be stable for 20 weeks in steel at 50 C, but in general the viscosity stability in contact with bare steel is not so good as the chemical or pressure stability of unthickened H. In glass, however, HV has shown a maximum of 33 per cent change in viscosity after 99 days at 65 C, and the stability in lined steel containers should be nearly as good. Thickened HD is somewhat better, but even this cannot be expected to retain its original viscosity for more than a few weeks in lacquered 75-mm shell at 65 C. Laboratory data[52] using coated steel test pieces providing the same ratio of metal surface to liquid volume showed excellent stability for three months at 65 C in nine out of ten samples of H and HD containing nitrogen bases and hexamethylene tetramine.

The available data appear to be inconclusive as to whether thickening at the plant is practical or not. The limit of stability of HV in lined M47 bombs is perhaps 3 to 6 months at 65 C if hexamethylene tetramine is used; this may correspond to anything from six months to several years actual storage. Certainly more conclusive stability data should be obtained before thickening at the plant is standardized, and it may well develop that more stable thickeners are required. In this situation a change from Levinstein-H to purified H is not the cure, since the storage life appears to be determined by the thickener and not by chemical stability or pressure development. The use of pure H, however, would simplify the search for a more stable thickening agent.

10.6 TACTICAL USE OF VESICANTS

The development of munitions and their tactical use has been primarily the responsibility of CWS, but practical problems relating to the use of vesicants have required laboratory investigations other than those outlined above. In addition to the critical reports of the joint CWS-NDRC Committee on the "Tactical Use of Vesicants," this section issued a general report on airplane vesicant spray.[25]

In the early stages of World War II the British recognized the greatly increased effectiveness of large drops for anti-personnel use of vesicants, and recommended the range of drop sizes from 1.5 to 5 mm. Essentially no drops larger than 1.0-mm diameter are produced in the case of H sprayed from any altitude. The vapor hazard of the H hitting the ground is not greatly dependent on drop size, but evaporation is more rapid from ground contaminated with fine spray. A heavy spray (greater than 5 grams per sq m) of unthickened H will produce heavy casualties among lightly clothed troops in the open, but it seems probable that larger drops are required to produce casualties among heavily clothed men. A number of field trials using spray against troops has been run both at Suffield and at Dugway, and a report[46] of the U. S. Chemical Warfare Committee concludes that unthickened H is satisfactory for low-altitude spray. However, the available field test data are inadequate to relate quantitatively the various factors such as contamination density, drop size, nature of clothing and protection, and probability of casualties.

In the case of spray from altitudes above 1,000 ft, thickening is essential to make aiming possible, and to reduce loss of vesicant by evaporation during fall. Experiments to determine rates of evaporation and terminal velocities were undertaken, but it was found that the literature was sufficiently complete so that relatively few new data were needed. Figure 3 shows the results of a general correlation[2] on the basis of which the rate of evaporation from spheres may be predicted. Here the ordinate is

$$j = \frac{k_c P}{u p_{BM}} \left(\frac{\mu}{\rho b} \right)^{2/3}$$

and the abscissa is the Reynolds number $Du\rho/\mu$ for the relative motion of a sphere through air. The symbols are defined as:

k_c = evaporation coefficient, gram mole/sec (sq cm) (gram mole/cu cm).

P = total pressure, atm.

p_{BM} = mean partial pressure of air in the gas film surrounding the sphere, atm.
u = velocity of sphere through air, cm/sec.
μ = air viscosity, poises.
ρ = air density, gram/cu cm.
b = diffusity of vapor in air, cm^2/sec.
D = drop diameter, cm.

In the case of H, the group $\mu/\rho b$ is approximately 2.5 and is independent of pressure. From the known vapor pressure of H (the wet bulb effect in cooling the drops is negligible), the rate of evaporation can be determined from this correlation for any diameter and velocity. Drag coefficients for spheres are well known,[2] and the correlations in terms of Reynolds number give reliable terminal velocities. Drag coefficients for the largest drops are larger than indicated by the general correlation, but these large drops are flattened and tend to break up. The largest stable drops are not spheres, but the correlations hold reasonably well. The nature of the calculated results, obtained by the method outlined, is indicated by the following values for per cent vaporization of pure mustard in falling from high altitudes.

TABLE 4. Per cent evaporation of pure *bis*-β-chloroethyl sulfide in fall from various altitudes. (20 C)

	Original drop diameter, mm				
Elevation, ft	6.0	2.0	1.0	0.5	0.3
1,000	0	1	2	12	42
5,000	1	4	13	59	100
10,000	1	8	28	100	100
20,000	1	16	56	100	100

Table 4 indicates the necessity of thickening in the case of high spray. Similar figures have been obtained by British workers. Vaporization of H is somewhat less than for pure mustard, since the nonvolatile impurities give a residue having some 70 per cent of the original drop diameter, and the velocity of fall is increased.

In order to determine the possible effect of thickening on rate of evaporation, data were obtained on weight loss of single drops suspended from a quartz spiral spring[52] hanging in a stream of air. For pure mustard and H containing 5 per cent chlorinated rubber no difference in rate was noted. A mixture of 47.5 per cent H, 47.5 per cent lewisite, and 5 per cent chlorobenzene likewise showed no appreciable effect of thickening with polystyrene, methyl methacrylate, or isobutyl methacrylate.

Penetration of cloth by drops of thickened and unthickened liquids was investigated by a photographic technique which gave pictures of the drop on impact after a free fall of 5 ft. With open fabrics, streamers of thickened liquid would protrude momentarily through the lower side of the cloth, only to be withdrawn immediately back into the fabric by surface tension. Closely woven fabric of either cotton or wool absorbs the drops completely without even momentary penetration. Silesia cotton lining, OD Pc.244, is an example of a borderline case showing slight momentary penetration. Figure 4 illustrates the effects obtained.

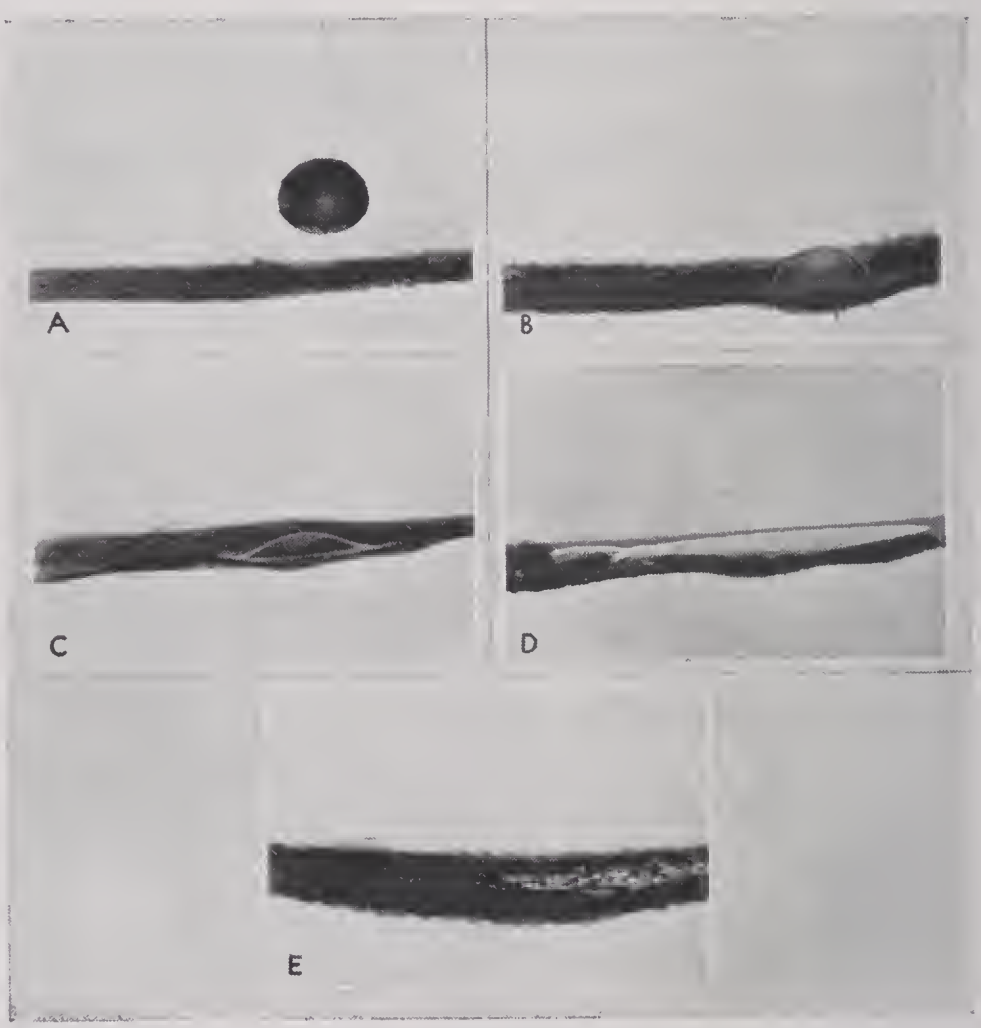

FIGURE 4. Absorption of a falling drop of liquid H by Silesia cotton lining, OD Pc 244.

10.7 CHEMICAL NATURE OF LEVINSTEIN-H

Research on the chemical nature of Levinstein-H fell within the province of Division 9. It may be noted, however, that work on this subject was carried out in connection with the studies of H purification and vesicant thickening.[19,52] Residues obtained by the molecular distillation of Edgewood Levinstein-H were shown to have chemical and physical properties essentially identical to those of HS_6.

10.8 LESS PERSISTENT VESICANTS

10.8.1 Introduction

Although mustard gas is one of the most effective and practical war gases, it has very important limitations. Since the vapors are readily adsorbed by activated charcoal, troops wearing masks may become casualties only by direct contact with the liquid or by extended exposure to the vapor. Offensive use of the vapor has not been generally practical because munitions for vapor generation have not been perfected, and evaporation of H deposited on soil or leaves is too slow to generate toxic gas clouds in a short time.

Dosage of H vapor is expressed in terms of the product *Ct*, where *C* is the concentration of H in air expressed as milligrams per cubic meter, and *t* is the exposure time in minutes. With masked troops at 60 F, the *Ct* must be 1,000 to 2,000 to produce a high proportion of casualties. The corresponding range for masked troops at 90 to 100 F is only 300 to 500. If a low-flying plane flying cross-wind contaminates a long, narrow strip of ground 50 yd wide to a density of 12 grams H per sq m, the ultimate *Ct* 100 yd downwind from the upwind edge of the strip is about 430, but a *Ct* of 320 is reached only after 2 hours. These estimates are based on Porton Memorandum 2515, assuming 2.0-mm drops, a wind velocity of 10.9 mph, and a ground temperature of 90 F. It is evident that masked troops might spend up to an hour or more in the area immediately downwind from such a spray deposit of H without becoming casualties.

Levinstein-H vapor is clearly more effective for offensive use in tropical than in cold climates, but an equally good vesicant of less persistence would have very much greater value. One approach is to develop a new agent having a greater vapor pressure than H, and this has, of course, been attempted. Still another approach is to modify H so as to cause it to vaporize more rapidly. This latter led to the development of the mixed agent known as HP, which, though not perfected, has shown remarkable promise.

10.8.2 Development of HP

The research under NDRC leading to the HP formulation was based on the idea that an additive might be incorporated into H, which, on exposure to the atmosphere, would react with the moisture or oxygen of the air to produce heat, thus raising the temperature and causing an increase in the rate of vaporization. In practically all the work the additive was white phosphorus [WP], which was employed in small amounts in H, and which was found to result in liquid temperatures as high as 190 C. The effect on the rate of vaporization of the active agent was, of course, enormous. Although the HP formulation was not developed to the point of adoption, the results obtained were quite promising. The basic idea seems applicable to other chemical warfare agents, and possibly for other purposes.

White phosphorus is soluble to the extent of 2 per cent at 20 C in either distilled or Levinstein-H. The amount of WP can be increased by the use of a cosolvent, but attempts to produce stable emulsions were not successful. The early compositions showing promise were (1) a straight 2 per cent solution, and (2) a solution of 4.0 per cent WP, 15 per cent carbon disulfide, and 81 per cent H. In laboratory tests the liquid surface was exposed to air at a measured velocity, and the rate of vaporization obtained by absorbing the H carried away by the air stream. It was found that composition (1) evaporated three to five times as fast as the H control, and the enhanced rate of evaporation of (2) was twenty to thirty times that of the control. The marked effect of the WP was largely confined to the first few minutes of exposure, however, and did not hold for the vaporization of more than a few per cent of the sample.

Various laboratory tests were designed to measure the effect of WP addition to H, but it became evident that the results could not be extrapolated to field conditions, since air velocity, nature of support for the liquid, and other variables, had important effects on the results. Accordingly, tests were run at Suffield in the fall of 1944,[44, 45] using both the annulus technique and static bomb bursts with H and with the two compositions noted above as (1) and (2). Vapor samples downwind showed the average vapor concentrations to be about 30 per cent greater in the case of the HP compositions than for straight H. This was true for the first hour; the effect of the WP tended to disappear with time. The bomb with the HP composition containing carbon disulfide inflamed.

Since the nature of the support for the liquid seemed so important, experiments with various supports were undertaken. Materials were chosen having thermal insulating value and large capacity to absorb liquid H. The results obtained were very remarkable. Using a ½-in. cube of regenerated cellulose sponge soaked in composition (2), it was found that the vaporization was very rapid, and that the center of the sponge reached temperatures as high as 190 C. The H evaporated amounted to 30 to 40 per cent in 300 sec, and careful chemical analyses proved that the H vaporized had not been decomposed. The H was generated as a dense smoke cloud, with a slight phosphorescent glow but no flame on the cellulose sponge. Similar results were obtained without cosolvent by dissolving 4 per cent WP in H at 50 C before saturating the sponge. Other materials were tested as supporting media, including glass fiber, Neoprene sponge, wood shavings, blotting paper, puffed rice, vermiculite, Santocel, cotton, and wool. The cellulose sponge with pores about 1-mm diameter seemed best, but was not chemically stable on storage in contact with H. The most promising support which was both effective and stable was wool in the form of fabrics or bats.

The use of carbon disulfide makes the wet sponges have a tendency to inflame, so a search was made for a nonvolatile cosolvent. Cyclic ethylene trithiocarbonate was found to be excellent, and was used in the composition (3): 81.5 per cent H, 3.5 per cent WP, and 15 per cent cyclic ethylene trithiocarbonate. This composition on ½-in. cellulose sponges vaporizes to the extent of about 50 per cent in 5 minutes, with copious clouds of H smoke, and the reaction starts immediately on exposure to air, with no induction period. Laboratory tests show the rates of vaporization in the early stages to be 3.5 to 10 times that of H, when the liquid is supported on soil or leaves instead of on sponges. Toy mortar shots showed no tendency of the atomized liquid to inflame. The vaporization of the liquid from soil or leaves in air at 20 C amounts to 1 to 3 per cent in 20 minutes, as compared to 0.3 per cent for H.

The problem of locating a suitable absorbent support in order to obtain the greatly increased vaporization rates has not been solved. Cellulose sponge is effective, but is not stable on storage with HP. The amount of the agent expelled as a liquid when a bomb explodes is probably excessive with materials such as cellulose sponge. Preliminary bursting tests have indicated that 18 to 37 per cent of the liquid is retained by Neoprene sponges. Combining the laboratory data on rates of vaporization of HP from sponges and from the liquid distributed on soil and leaves with this figure for liquid retention on explosion, it is estimated that the total H vaporization in 400 sec should be 3 to 5 per cent, as compared with 0.07 per cent in the same time period for H. Under conditions where a lethal *Ct* of 1,000 to 1,500 is obtained in 2 to 3 hours with H, the same *Ct* should be obtained in a very few minutes, using bombs charged HP on porous supports.

The basic idea of HP should be applicable to other agents. WP is soluble to the extent of 1.2 per cent in HN-3, and the use of cosolvents should make it possible to increase this concentration. WP appears to be the best additive, but others are possible. Trialkyl borines, stibines, and arsines provided by Division 9 were tested with H, but showed little promise.

Chapter 11

EXTERIOR BALLISTICS OF LIQUID-FILLED SHELLS

11.1 SUMMARY

The complicated problems of the exterior ballistics of liquid-filled shells have been attacked, with special reference to the 4.2-in. chemical mortar shell, by (1) a study of the existing theory and data on liquid-filled shells, (2) experiments with a liquid-filled spinning top, using cavities of three shapes, liquids of various densities and viscosities, and internal vanes of several types, (3) field trials with 1-in. models of the 4.2-in. mortar shell, and (4) the design and construction of a laboratory ballistics testing machine in which full-sized shells supported on gimbals may be subjected to the various forces simulating flight along a curved trajectory. The testing machine has been completed and experiments with it will be made under a continuation of the contract by CWS.

The proof-firing data and the results of the tests with the 1-in. models indicate that the 4.2-in. shell may be stable near the gun, but develop instability at or beyond the vertex caused either by excessive spin (excessive stability) making the shell fail to follow the curved trajectory, or by excessive yaw developing near the vertex, which causes the shell to tumble. The experiments with the top indicated the effect of internal vane design in causing erratic nutational motion in a shell which was stable in the ordinary sense. Large momentary yaws developed at speeds well above the critical with certain vane designs, and this effect may well explain tumbling of shells with high stability factors.

11.2 INTRODUCTION

There exists a very considerable background of theory and experience relating to the flight characteristics of solid shells, and their trajectories may be predicted with a fair degree of certainty. Motion along flat or curved trajectories may be calculated, provided the angle of yaw does not become greater than about 15 or 20 degrees. Conditions for stable flight of liquid-filled shells are but partially understood, and the problem has become urgent because of the erratic behavior of some of the important chemical munitions. Liquid filling complicates the theory of solid shells by introducing such factors as liquid viscosity, liquid density, and internal baffle vane design.

The motion of a solid spinning projectile is similar to that of a top spinning on a fixed pivot.[11,12] The spin causes the shell to retain its general orientation with respect to the earth, but a precessional motion develops which causes the angle of yaw to increase and decrease. As yaw develops, the air pressure causes a force to be applied to a center of pressure ahead of the center of gravity, and an overturning moment develops which may increase the yaw and cause the shell to tumble. The condition for stability at small angles of yaw is

$$\frac{A^2N^2}{4B\mu g_c} = \text{Stability factor} > 1.0$$

where A = moment of inertia about the spin axis.
B = moment of inertia about the transverse axis through the center of motion.
N = spin velocity.
μ = overturning moment coefficient (overturning moment $= \mu$ times sine of angle of yaw).
g_c = gravitational conversion factor.

Shells are usually designed so that the stability factor will be greater than 1.4. If it is much above 2.0, the stability may be excessive, causing failure of the shell axis to follow the curved trajectory. This is especially noticeable at high angles of fire, at which shells with high spin "sit down" on their bases, because they maintain too closely their original orientation with respect to the earth.

Data on firing trials with liquid-filled 75-, 105-, and 155-mm shells, made available by the Ballistics Research Laboratory at Aberdeen, show no important differences between the results with HE and liquid fillings. The stability problem in the case of the 4.2-in. mortar shell is quite different, however, since the ratio of liquid weight (H-filled) to total filled weight is 0.26, as compared to ratios of 0.075, 0.097, and 0.124 for the 75-, 105-, and 155-mm shells, respectively. Certain types of the 4.2-in. shell have been found to be stable[4] when employed with solid filling, but unstable with liquid filling. Such shells (E49R1) without internal vanes have been found stable when filled with 7.0 lb solid WP, but unstable when filled with 7.0 to 7.1 lb liquid chlorosulfonic acid. When the liquid content was reduced to 5.8 to 6.0 lb, the shell became stable. That the internal vane has a stabilizing influence is indicated by the fact that a vaned shell (E38R2) containing 7.8 lb chlorosulfonic acid was stable. Almost no proof-firing data exist as a basis for demonstrating the effect of the viscosity of the liquid filling. The effect of void space is apparently complicated, as the proof-firing data relating to this variable are quite contradictory.[5,6,13,14,15]

The most advanced analytical treatment of the exterior ballistics of liquid-filled shells is that of E. A. Milne.[5] Milne's theory is developed on the basis of a projectile with an ellipsoidal cavity having its center coincident with the center of gravity of the shell, filled with an ideal liquid of zero viscosity. Two dimensionless parameters are developed which divide stable and unstable shells, assumed to be following a flat trajectory with negligible overturning moment. In spite of the simplifying assumptions introduced, the two parameters were found to serve as an empirical basis of distinguishing stable and unstable designs of actual shells. As applied to United States shells, the method indicates that the 105- and 155-mm shells should be unstable without burster wells. The Milne criterion of stability apparently does not apply in the case of shells such as the 4.2-in., which has a high ratio of weight of filling to total filled weight, and which has a burster well and internal vanes. The Milne theory, however, is being extended by the staff of the Ballistics Research Laboratory to allow for flight along a curved trajectory, and for cases where the centers of gravity of shell and filling do not coincide.

It has been pointed out[a] that the observed instability of the 4.2-in. shell may develop at the vertex of the trajectory, beyond the region in which the Milne theory applies. As the curvature of the trajectory increases rapidly, there develops a steady yaw away from the plane of the trajectory, and calculations indicate that the yaw of the 4.2-in. shell near the vertex may become quite large. It is suggested that erratic internal liquid motion or negative aerodynamic damping may cause instability to develop at or beyond the vertex, even though motion along a straight trajectory is stable.

11.3 EXPERIMENTS WITH A LIQUID-FILLED TOP

Because of the similarity of the gyratory motions of a spinning shell in flight and a top spinning on a fixed pivot, laboratory experiments with a liquid-filled top were undertaken. The top consisted of a cylindrical steel case 5⁹⁄₃₂ in. long by 4³¹⁄₃₂-in. OD having a removable cover and pivot points on both ends. Three removable inserts were provided with cavities of three types. One was a 4½-in. spherical cavity, one was an ellipsoid with a ratio of major (axis of revolution) to minor axis of 2, and the third was a cylinder with internal length and diameter both 4½ in. Liquids of various types and in varying amounts were employed in the three cavities.

The experimental top was started spinning on fixed top and bottom bearings. The upper bearing was then removed and the motion observed as the speed of revolution slowly decreased due to air and bottom bearing friction. The speed was observed at intervals by means of a Strobotac until the critical speed was reached, when the top became unstable and fell over. Internal vanes of various designs were tested using the spherical and the cylindrical cavities. A short length of ⅝-in. OD copper tubing on the vertical axis was introduced to simulate a burster well.

The critical speed of a solid or empty top is readily calculated from its dynamical constants. The expression for the critical speed of a liquid-filled top is likewise easily obtainable if the liquid is assumed to slip freely at the solid-liquid boundary. Figure 1 illustrates the two theoretical relations for the top having a spherical cavity filled with varying weights of liquids. The experimental points for several liquids are seen to fit the upper theoretical curve based on the assumption that viscosity is not important. The critical speed with the most viscous fluid (37 centipoises) is slightly less than the theoretical value, indicating a slight effect of viscosity in increasing stability.

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[a] J. J. Stoker of the Applied Mathematics Panel.

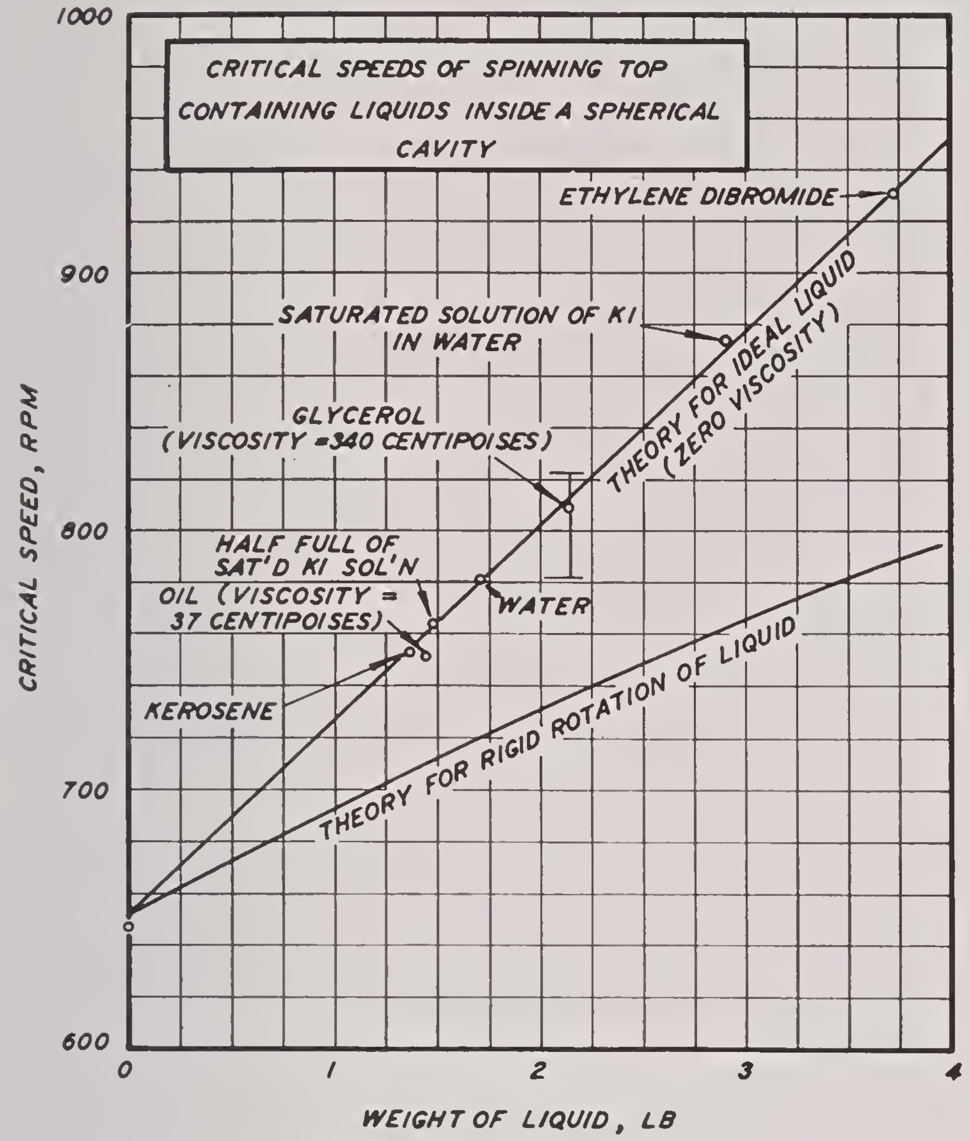

FIGURE 1. Critical speeds of spinning top containing liquid inside a spherical cavity.

The tests with glycerol in the spherical cavity gave peculiar results. Not only was the critical speed less reproducible, but also the normal smooth precessional motion at speeds above the critical was interrupted by short periods during which the amplitude increased suddenly, giving violent reactions at the lower bearing. When the cavity was $\frac{1}{6}$-full of mercury, it could not be brought to full speed because of excessive vibrations, suggesting that the mercury might be concentrated on one side of the cavity.

Similar tests with the ellipsoidal cavity gave results which checked the theory for rigid rotation, indicating that the elongated ends of the cavity tend to force the axis of the spinning fluid mass to tilt with the axis of the top. Erratic results were obtained with mercury, the motion consisting of alternate quiet periods of about 10 sec duration and brief periods of violent vibration. In the latter, intense lateral reactions developed at the bearings, and the top tended to jump out of the lower bearing.

Using the spherical cavity and vertical single or double (cross) vanes, the critical speeds checked the theory for rigid rotation of the liquid. Similar results were obtained with and without holes in the vanes, but the precessional motion was smoother without the holes, and the spin velocity decreased more rapidly when the vanes with holes were used.

Tests with the cylindrical cavity and vanes located parallel to the spin axis were run with varying fractions void. The effect of void fraction was found to be quite complicated and to vary markedly with the vane design. In the vicinity of 10 per cent void the critical speed was 2,000 rpm, yet with the central tube only and no vanes the critical speed was 800 rpm.

The first conclusion from the experiments with the top is that the liquid does not follow the motion of the cavity and that it is safe to draw a similar conclusion for the flight of an actual shell. Perhaps the most important result of the top experiments, however, was to demonstrate the erratic nutational motion frequently obtained at speeds well above the critical, when the motion is stable in the ordinary sense. In the case of artillery shells, the simplified theory of stability—the analog of the exact theory for the top—fails when the yaw becomes large, and experiment indicates that a shell becomes unstable whenever the yaw becomes large. Erratic behavior of a liquid-filled shell, similar to the violent behavior of the liquid-filled top, may cause the shell to become unstable even though the analogous motion of the top is still stable in the sense of continuing to spin at velocities well above the critical.

The erratic behavior at speeds above the critical was common in the case of vanes set parallel to the spin axis, and there was a wide variation of critical speed with void fraction. In the case of single vanes, the vane is presented alternately broadside and edgewise to the fluid when the top yaws, and the top behaves as though its moment of inertia depended on its orientation with respect to the spin axis. The tests with varying void spaces indicated that best results would be obtained without vanes. This conclusion is not applicable to a shell, however, as the analogy is incomplete, especially for high-angle fire, since it is desirable that the liquid's spin axis as well as the shell's axis turn at the same rate as the tangent to the trajectory.

As a result of an analysis of the problem based on these tests, the recommendation was made that the 4.2-in. shell be fitted with a number of perforated circular disks within the liquid cavity, spaced at intervals and perpendicular to the axis. The purpose would be to divide the liquid into small masses, each capable of turning independently of the others. A schedule of field trials of 4.2-in. shells with vanes of this type was set up, but the results are not yet available. Top experiments with vanes of this type gave the most stable motion of any arrangement tried, and greatly reduced the critical speeds for intermediate void fractions.

11.4 EXPERIMENTS WITH 1-IN. MODELS OF THE 4.2-IN. CHEMICAL MORTAR SHELL

In connection with the study of vane design in the 4.2-in. mortar shell a series of tests was carried out using a small model mortar at Aberdeen Proving Ground. This experimental mortar was designed to shoot 1-in. projectiles at high angles with a charge consisting of a few grains of black powder. Twelve scale models of the 4.2-in. shell were constructed with various types of internal vane construction. The empty shells weighed 146.1 to 167.0 grams and the volume of the cavities varied from 29.6 to 32.4 cu cm. Four mortar barrels were used, having one rifling twist in 15, 20, 25, and 30 calibers, respectively. The charge of 5 grains employed was sufficient to give a range of 241 ft with a 190-gram projectile fired at an angle of 40 degrees. The calculated muzzle velocities varied from 118 to 156 fps. Several liquid charges were used, with various void fractions. A motion picture camera was employed to follow the shells in flight.

With a shell 88 per cent full of ethylene dibromide and fitted with a vane of standard design and a barrel having a rifling pitch of one in twenty, large yaws were obtained on the far end of the trajectory beyond the vertex at all elevations above 32 degrees. True flight was obtained with the same shell up to angles of 45 to 50 degrees when the rifling twist of one in twenty-five was used. The same shell filled with tetrabromethane (specific gravity 2.96) showed completely unstable behavior with void spaces of zero and 17 per cent, but the instability disappeared when water was used instead of the heavy liquid. A large increase in the viscosity of the charge (to 20 poises) showed some indication of giving better stability with the standard 4.2-in. vane design. The effect of rifling twist was clear: at pitches of 1:15 and 1:20 instability developed beyond the apex; best results were obtained with a pitch of 1:25; with a pitch of 1:30 excessive yaw developed near the gun.

The variables having the most pronounced effect on stability were the rifling pitch, the angle of elevation, and the muzzle velocity. The use of vanes of various designs changed the results very little. Decrease in liquid density or large increase in viscosity improved stability. In most of the experiments the observed instability was actually overstability, since the yaws were steady and to the right of the trajectory. Departure from true flight occurred when this steady yaw failed to disappear after the shell had passed the top of the trajectory. Instability of this type could be corrected by changing the rifling of the barrel to reduce spin, or by increasing the muzzle velocity. Neither of these solutions to the problem of the 4.2-in. mortar shell were acceptable in time of war.

11.5 LABORATORY BALLISTICS TESTING MACHINE

Experimental ballistics studies in the field have proved extremely helpful in connection with many important problems, and the technique has been developed to an advanced stage.

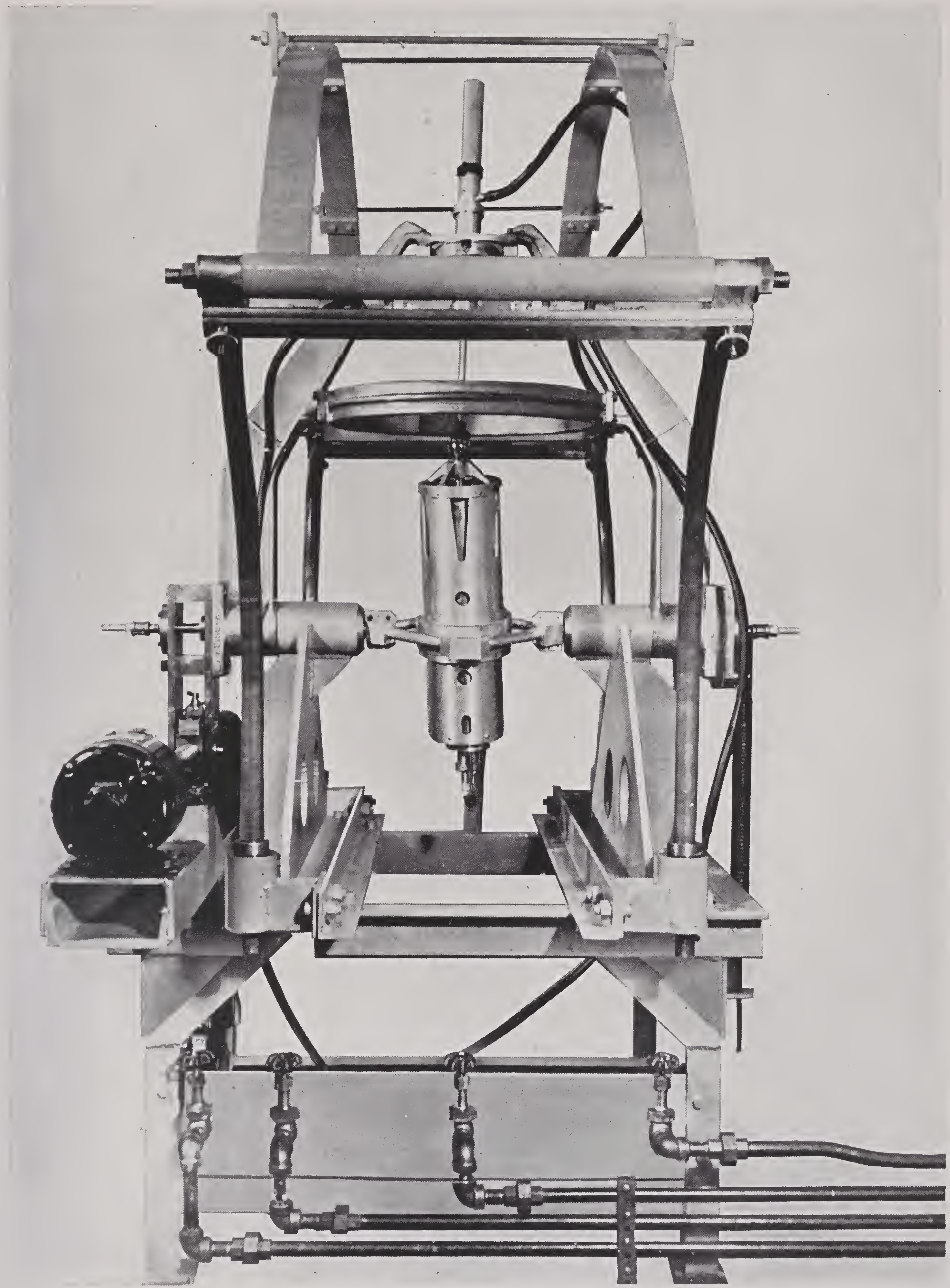

FIGURE 2. Laboratory ballistics testing machine.

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Such tests are time-consuming and expensive, and require statistical analyses of large numbers of observations. For these reasons, it is exceedingly difficult to study the separate effects of the many variables affecting the stability of liquid-filled shell, and a practical laboratory test technique would prove extremely useful.

There appear to have been three[5,12,13] previous attempts to simulate the motion of a shell in flight by spinning a shell or shell model supported in gimbals so as to be free to turn in any direction about its center of gravity. These were not carried to the point of making careful measurements, and it appears that in none of the three cases were external forces applied simulating the forces acting on the shell in flight.

With the encouragement of CWS and of the Ballistics Research Laboratory at Aberdeen, a laboratory testing machine was designed and constructed with the object of observing and measuring the nature of motion of full-sized shells, such as the 4.2-in. mortar shell, when rotated at high speeds and subjected to specified forces simulating the varying overturning moment along a curved trajectory. A total of fourteen preliminary designs were prepared before the final design was agreed upon. Figure 2 illustrates the test machine as finally constructed.

The 4.2-in. chemical mortar shell which forms the rotor is fitted with a special base and nosepiece which allow it to turn in ball bearings. The internal design is not altered, and the burster well is in place. The tube which encloses the shell, the gimbal ring, and other moving parts are made of aluminum. The total transverse amount of inertia of the entire assembly is 745 (lb mass) (in.^{2}), as compared with 546 for the original shell. The moment about the spin axis is 40.7 (lb mass) (in.^{2}), as compared with 47.0 for the 4.2-in. shell. Air jets and small nozzles in the aluminum cage are used to spin the shell, and a second manifold and set of nozzles is used to stop the spinning shell after a test. At the top is an air piston designed to apply a thrust to a push rod attached to ball-and-socket joint on the nose of the shell, simulating an overturning moment approximately proportional to the sine of the angle of yaw. This air piston is moved slowly in an arc from front to rear of the figure, this motion approximating the varying direction of the overturning force as the shell follows a parabolic trajectory. The lead screw which operates the traversing drive rotates at a constant speed during an experiment. The crank and sliding nut arrangement is designed to obtain an angular velocity of the frame which is proportional to the square of the cosine of the angle which the frame makes with a vertical plane. This variation of angular velocity is that of a tangent to a parabolic trajectory. The time of movement from an initial position 45 degrees to the vertical on one side to the final position 45 degrees on the other side corresponds to the time of flight of the shell and can be varied.

A record of the shell's motion is made by means of an optical system carried by the overturning moment applicator. An interrupted light source produces a record on a sheet of photographic paper laid across the arch above the machine. This gives a record of the precessional motion of the nose of the shell. The rotational speed of the shell is measured by means of a specially constructed stroboscopic device.

The principle of operation is to operate a spinning shell under conditions simulating flight along a curved trajectory, with the aerodynamic forces acting on the shell in flight simulated by a single overturning moment porportional to the sine of the angle of yaw. The curved trajectory is reproduced by having the point at which the overturning moment applicator is pivoted move about the shell's center of gravity at a rate equal to the angular velocity of a tangent to the actual trajectory. Figure 3 illustrates the nature of the data obtainable with the test device described. Owing to the method of starting, this first test result is not typical of a shell in flight, since the nutational motion shown is increasing.

At the termination of the contract in the fall of 1945, the machine had been constructed but no experiments had been made with it. The investigation is being continued under a contract with CWS.

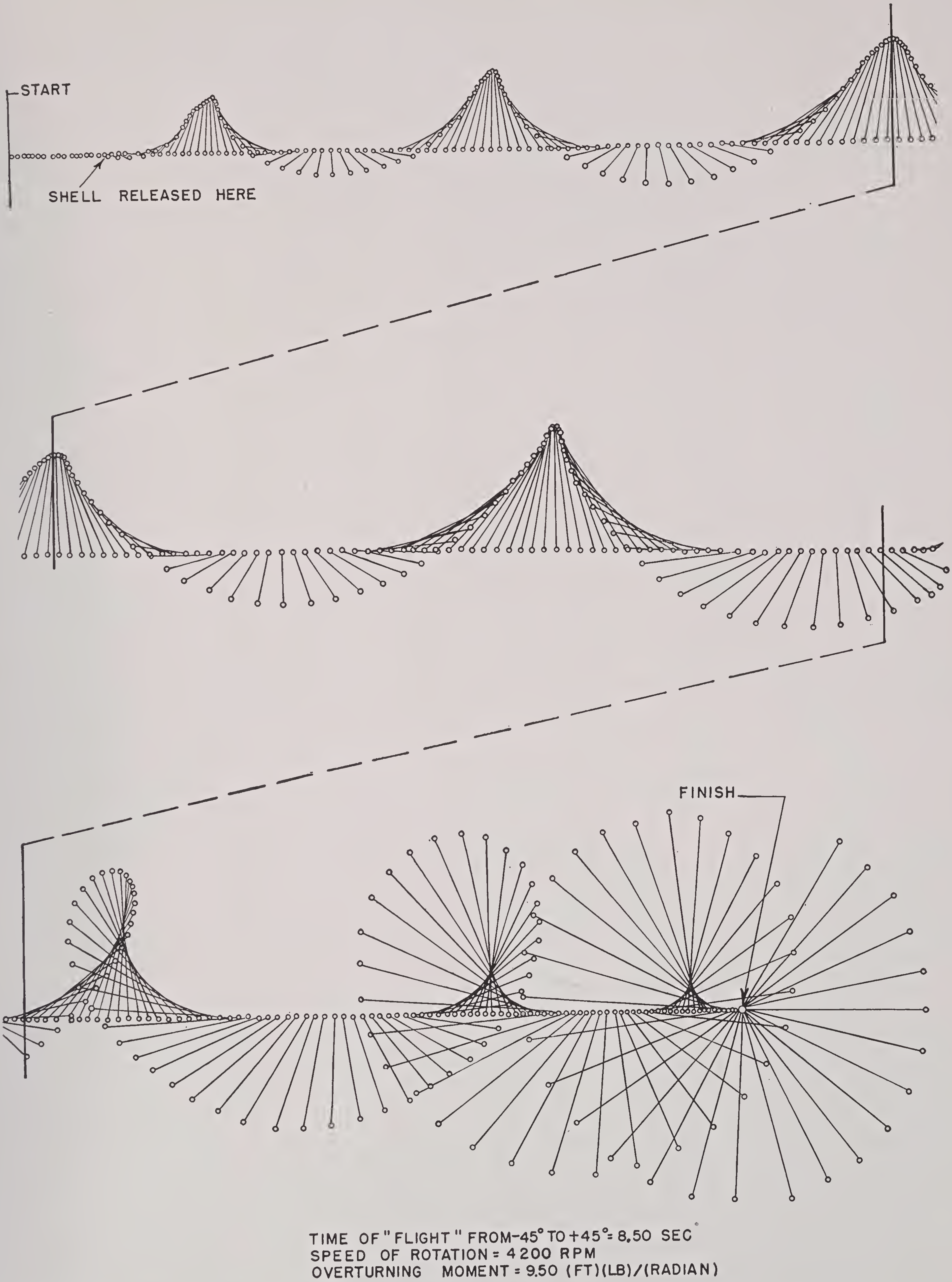
START
SHELL RELEASED HERE
FINISH
TIME OF "FLIGHT" FROM -45° TO +45° = 8.50 SEC
SPEED OF ROTATION = 4200 RPM
OVERTURNING MOMENT = 9.50 (FT)(LB)/(RADIAN)

FIGURE 3. Motion of empty shell observed experimentally. (Curved locus of points represents motion of nose of shell as viewed end-on.)

Chapter 12

HYDROGEN GENERATOR FOR PRESSURIZING PORTABLE FLAME THROWERS

12.1 SUMMARY

THE USE OF COMPRESSED nitrogen for pressurizing portable flame throwers has the great disadvantage of requiring a battery of four nitrogen cylinders weighing 600 lb. The small air compressor for the purpose weighs 820 lb, and neither the cylinders nor the compressor are readily transported to the front lines where the flame throwers are used.

As an alternative to these methods, a high-pressure hydrogen generator weighing 49 lb was developed and procurement started in the spring of 1945. Hydrogen was generated by the chemical reaction of water and lithium hydride. For pressurizing one M2-2 flame thrower, the chemical charge consisted of 0.5 lb granular lithium hydride in a container weighing 1.2 lb filled, and the salt or fresh water required for reaction and cooling weighed 2.2 lb.

12.2 INTRODUCTION

Fuel is ejected from the fuel tanks of portable flame throwers by compressed air or nitrogen from the pressure tank of the flame thrower. These pressure tanks are filled to a pressure of from 1,700 to 2,000 psi from a battery of large commercial nitrogen cylinders, or by the use of truck-mounted air compressors weighing 820 lb. This servicing must of necessity be done at a considerable distance behind the front lines, seriously limiting the number of missions per day of a given flame thrower. The desirability of a lightweight generator, capable of refilling the pressure tanks of portable flame throwers near the point of discharge, is obvious. In addition to the time saved, the need for the transportation of a large amount of heavy equipment, per pound of available gas, is eliminated.

Consideration of various methods of generating gas under pressure led to selection of two alternatives: the reaction of a metal hydride with water, and the controlled burning of double base powders. Development of the second alternative was undertaken by the Allegany Ballistics Laboratory of NDRC Division 3, and is reported elsewhere.[7] Good results were obtained by the first method using lithium hydride, calcium hydride, sodium hydride, and a mixture of sodium hydride and aluminum (see Chapter 7); but the first was selected because of its availability and light weight, and because it showed no tendency to catch fire on exposure to the atmosphere.

Several experimental hydride generators were constructed and tested at Edgewood Arsenal in October 1944. The most promising was developed further and became the E5, of which twenty-five were procured and delivered for test by the Infantry Board,[2] the Marine Equipment Board,[5] the Airborne Board,[3] and the Chemical Warfare Board.[1,4] Adoption was recommended in each case, and final drawings of the E5R1 were prepared by the CWS Development Laboratory. These generators were in large-scale production at the end of World War II.

12.3 OPERATION OF THE GENERATOR

The E5R1 generator is illustrated in Figure 1.[6] In operation of the generator, a cartridge containing lithium hydride is loaded into the breech of the reaction chamber, and a separate bottle is filled with water. The water used to react with one cartridge is approximately 1.2 lb. The water flows by gravity into the reaction chamber where it reacts with the cartridge. The generated hydrogen flows through cooling coils which lower the temperature of the gas to within 10 or 20 F of ambient temperature. The entrained water is removed from the gas stream by a cyclone separator at the

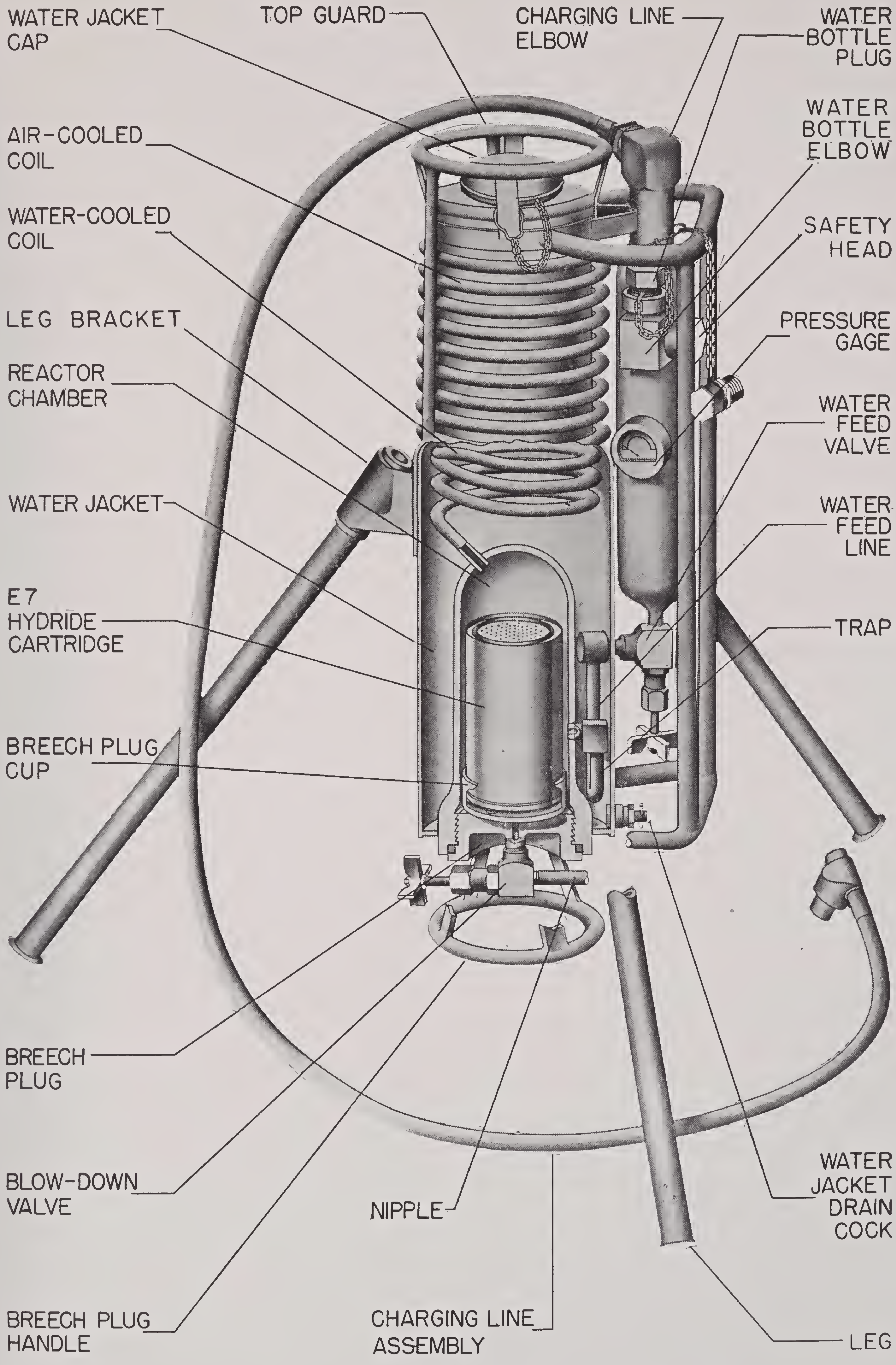
WATER JACKET CAP
TOP GUARD
CHARGING LINE ELBOW
WATER BOTTLE PLUG
AIR-COOLED COIL
WATER BOTTLE ELBOW
WATER-COOLED COIL
SAFETY HEAD
LEG BRACKET
PRESSURE GAGE
REACTOR CHAMBER
WATER FEED VALVE
WATER JACKET
WATER FEED LINE
E7 HYDRIDE CARTRIDGE
TRAP
BREECH PLUG CUP
BREECH PLUG
BLOW-DOWN VALVE
NIPPLE
WATER JACKET DRAIN COCK
BREECH PLUG HANDLE
CHARGING LINE ASSEMBLY
LEG

FIGURE 1. E5R1 hydrogen generator assembly for pressurizing flame throwers.

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top of the water bottle, and the cool dry gas then flows through a charging hose to the pressure tank of the portable flame thrower. When the desired pressure is reached, the excess water and hydrogen are released from the generator. The breech is opened, and the spent cartridge is removed and discarded. The bulk of the hot hydroxide residue stays in the cartridge so that disposal is safe and convenient. According to the Marine Board,[5] an average Marine requires 25 minutes to learn how to run the E5 generator. By following the directions on the instruction plate attached to the generator, 4 to 6 cycles can be made per hour. With repeated runs, a maximum of 1 pound of water is evaporated from the jacket in each run; with infrequent runs little or no make-up cooling water is required. In emergencies, sea water may be used both for reaction and cooling.

The final cartridge (E7 hydride cartridge) contains 0.5 pound of 14- to 18-mesh (U. S. standard screens) lithium hydride in a perforated steel basket surrounded by a tinned steel sleeve open at both ends, the whole shipped in a key-opening, airtight can.

The E5 generator was subjected successfully to various rough handling tests, including parachute drops from an elevation of 800 ft. The Boards reported that the E5 generator was easily carried on packboard by one man over any terrain accessible to a man carrying a portable flame thrower. A second man carried the necessary supply of cartridges and extra water.

The dimensions and weights of the E5 generator are summarized in the following table.

Weight generator, empty	49 lb
Loaded, water in jacket, ready to run	59 lb
Empty, in packing chest (16½ x 15 x 27½ in.) with tools, spare parts, and accessories, packed for overseas shipment	100 lb
Wooden box containing 36 lithium hydride cartridges (net weight 43 lb) ready for overseas shipment	78 lb
Maximum height generator, legs retracted	24½ in.
Maximum width generator, legs retracted	10 in.
Maximum diameter, legs retracted	12 in.
Maximum height, legs extended (operating position)	32 in.

In connection with the tests by the four Boards, and in auxiliary tests at MIT, nearly 1,000 chemical charges were fired in E5 generators. In subsequent tests, hundreds of E7 cartridges were fired in a single E5 generator, all without difficulty. Portable flame throwers, both M1A1 and M2-2, were successfully operated with hydrogen produced in E5 hydrogen generators, using both thickened and unthickened fuel. The results equal those obtained with nitrogen or compressed air, and hydrogen has the advantage over compressed air because it does not form an explosive mixture in the fuel tanks of flame throwers.

Table 1 summarizes the requirements for repressurizing M2-2 portable flame throwers. A comparison is made of the E5 generator and the conventional system that uses either cascade filling from large commercial nitrogen cylinders or repressurizing using a motor drive air compressor.

TABLE 1. Requirements for repressurizing M2-2 portable flame thrower.

Basis: 20 M2-2 flame thrower pressure cylinders repressurized per hr to 2,000 psi.

	Using nitrogen cylinders	Using E5 portable hydrogen generator	Using portable compressor*
Weight of unit pressurizing device,† lb	610‡	49	820
Number required	1	4	1
Total weight of pressurizing equipment, lb	610	196†	820
Weight of chemicals and containers shipped overseas, lb	400‡	24	5
Weight of water for reaction, lb	0	24	0
Weight of water for cooling, lb	0	20	0
Total weight of supplies expended, lb	400	68	5
Total weight of gas in repressurized cylinders, lb	22	2	23
Distance flame thrower pressure tank must be carried to be filled	Substantial	Small	Substantial
Pilot hydrogen supply for M1A1 flame thrower	Note 1	Note 2	Note 1

* Compressor, air, gasoline engine driven, 7 CFM, M1.

† If weights of crates, or volumes of shipments were included, the comparison would be even more favorable to the portable hydrogen generator.

‡ This figure assumes that four 150-lb nitrogen cylinders in a cascade filling system are used as per the "M2-2 Flame Thrower Manual" (TM 3-376A).

Note 1. Requires large commercial hydrogen cylinders, an additional item of supply, or else spare small hydrogen tanks.

Note 2. Small hydrogen igniter bottle is filled simultaneously with main pressure cylinder.

12.4 DESIGN FEATURES

12.4.1 Particle Size of Lithium Hydride

If the grains of LiH are too small, the rate of reaction is too rapid, seriously complicating the problems of heat dissipation and removal of mechanically entrained liquid and alkaline residues from the gas produced. With excessive fines, the resulting rapid hot reaction leaves molten residues which may plug valves or cooling coils while the surplus gas is being blown off at the end of a cycle. If the grains are too coarse, chemical reaction is incomplete, leaving a core of unreacted LiH at the center of the oversize grain. It was established that the optimum grain size is 14 to 18 mesh on U. S. standard screens. Cartridges containing the recommended range of grain sizes were subjected to standard rough handling tests and found to function properly in the generator.

12.4.2 Ratio of Water to Hydride

With the standard charge of 0.5 lb of lithium hydride, the stoichiometric quantity of water for complete reaction is 1.14 lb (based on reaction to LiOH). Best results were obtained with 107 to 112 per cent of the theoretical, namely 1.21 to 1.27 lb.

12.4.3 Cartridge

Two types of cartridges were developed. In the first, large area for water access was obtained by charging the hydride granules in a cylindrical basket made from 20-mesh copper screen with soldered seams at top, side, and bottom. This basket was surrounded with a tinned steel sleeve closed at both ends by taped-on paper covers, and this cartridge (sleeve. basket, and hydride) was packed in a key-opening airtight can. In use, the solder melted and at times bonded the spent cartridge to the breech plug, requiring use of a chisel, or caused the breech threads to become jammed.

The improved and final cartridge E7 substituted a perforated steel basket with crimped seams for the soldered copper screen basket described above. This was contained in a tinned steel sleeve having ½-in. rings at each end serving to keep the basket in place. This cartridge eliminated solder troubles, minimized leakage of molten residue, and combined better strength with ease and cheapness of production.

12.4.4 Breech Plug Cup

To prevent plugging of the blowdown valve (which is at the base of the breech plug), jamming of breech threads by solidification of previously molten residues, or sticking of spent cartridges in the reactor, a breech plug cup[6] was adopted.

12.4.5 Water Feed Time

The water is introduced by gravity from a water bottle which holds the recommended amount of water when filled to overflowing. The size of the water feed valve controls the time taken to empty the water bottle. If the opening in this valve is too large, the reaction will be so rapid that an excessively long air-cooled coil will be needed to cool the gas to within 10 to 20 F of ambient temperature, and difficulties with molten residue might be encountered. Very slow introduction of water limits the number of cycles per hour. Tests showed that good results were obtained with a water bottle drainage time of 50 to 70 sec, obtained with the standard ¼-in. stainless angle needle valve used on the E5 generator.

12.4.6 Cooling Requirements

During the gas generating period approximately 1,000 Btu per cycle are absorbed by the water jacket. With infrequent cycles, the jacket water never boils and makeup water is not required, but with 4 to 6 cycles per hr a maximum of 1 lb of jacket water is evaporated in each cycle. The bulk of the heat in the gas leaving the reactor is removed by three turns of helically coiled ⅜-in. OD Monel or Inconel tubing submerged in the water jacket, and the remainder is removed in nine turns of the same coil exposed to air outside the water jacket. During the blowdown period a substantial amount of heat is present in steam and surplus hydrogen gas bled from the blowdown valve at the base of the breech plug. A relatively large quantity of heat is removed with the spent cartridge as sensible heat (molten residue in the center of the can at approximately 1,000 F) and as chemical heat in the residue containing both LiOH and Li_2O.

12.4.7 Cyclone Separator

Even with the dome at the top of the reactor which serves to disengage foam from gas, some 10 ml of liquid is carried through the coils to the top of the water bottle, and is removed by the cyclone-type separator in the top of the water bottle. The liquid recirculates by gravity through the water feed valve. The gas is forced to flow through the cooling coils and cyclone separator, rather than through the water feed line, by installing a vertical U trap (2 in. high) in the water feed line.

12.4.8 Materials of Construction

At the request of the CWS, mild steel was used for the reactor and water bottle, but Monel or the equivalent was permitted for the cooling coil, water jacket, and certain small threaded parts, and stainless steel was authorized for valves. The reactor was designed with a wall $\frac{5}{16}$ in. thick, which includes a corrosion allowance of $\frac{1}{16}$ in.; the outer surface was copper plated in the case of the twenty-five E5 generators and was zinc plated on the E5R1 generators.

12.4.9 Safety Heads

The E5R1 contained one replaceable Kidde safety head, bursting in the range 2,500 to 3,000 psi.

12.4.10 Pressure Tests

Both an E5 and an E5R1 generator were pressure tested hydraulically. The pressure gauge on the generator operated successfully after being subjected to a pressure of 4,500 psi, and the Bourdon tube (in the gauge) ruptured at 9,500 psi. Upon blanking off the pressure gauge, the breech enlarged enough at 10,000 psi to allow the Neoprene breech gasket to extrude, releasing the pressure.

12.5 POSSIBLE FUTURE IMPROVEMENTS

At the time of the demonstration of the four types of generator at Edgewood in October 1944, the air-cooled unit shown was not made by a standard manufacturing operation, and hence the water-cooled unit was adopted. However, while the E5 and E5R1 developments were under way, two air-cooled units were developed, involving standard methods of manufacture. The Al-Fin type was prepared by casting aluminum alloy around the reactor and milling vertical fins, so that air would flow upwards between the fins by natural convection circulation. The Harrison Radiator type bonded copper to the reactor in the form of U-draped ribbon. In either event the fins were housed in an air jacket connected to a chimney, and the gas was cooled in a helical coil wound around the chimney. These two air-cooled reactors were assembled as experimental generators by Artisan Metal Products. Since they became available only at the time the project closed, they were turned over to the CWS Development Laboratory at MIT for testing and further development. The Harrison-Artisan air-cooled generators weighed approximately 49 pounds including the charging line, as compared with 58 pounds for E5 or E5R1 with full water jackets, and did not require cooling water, thereby reducing the total maximum water consumption of the water-cooled unit by 45 per cent.

The alternative development of a gas generator based on the burning of cordite or double base powder was carried to the point of successful demonstration of prototype models at Allegheny Ballistics Laboratory. The cordite generator was not developed far enough to justify definite conclusions regarding its relative merits as compared with the hydride generator, and it is possible that a very simple and lightweight cordite generator could be designed.

Chapter 13

INSTANTANEOUS RESPIRATION RATES

13.1 SUMMARY

INSTANTANEOUS AIR flows for inspiration and expiration were obtained for a wide range of work rates, environmental conditions, respiratory resistances, and types of work, by means of a new type of instrument. Instantaneous inspiration rates as high as 276 liters per minute were recorded for men doing heavy work. The use of a device simulating human breathing was developed for testing protective equipment, and the adoption of this technique resulted in the rejection of large quantities of charcoal found acceptable on the basis of the earlier breakdown test at a constant air rate of 32 liters per minute.

Fundamental data on the design, performance, and testing of outlet valves were obtained. Values for maximum allowable resistance for inspiration and for expiration were determined for medium and heavy work rates. It was found that the maximum permissible breathing resistances corresponded to breathing work of about 1.2 per cent of the external work being performed.

A portable instrument for measuring air flow under field conditions was developed and used to obtain data on respiration rates of men performing various tasks.

13.2 INTRODUCTION

In the early stages of World War II gas mask adsorbents and canisters were tested for protective efficiency and breakdown at a steady air flow of 32 liters per minute. Resistance of masks, valves, and canisters were evaluated at a steady air flow of 85 liters per minute. No critical evaluation of respiratory resistance existed upon which the upper limit for inspiratory canister and expiratory valve resistances could be specified.

A first objective was to determine the instantaneous rates of inspiratory air flow with subjects performing varying amounts of work. In order to obtain these values, it was necessary to use an instrument with negligible lag and inertia which would not add any significant resistance to breathing. Such a flow meter was developed for instantaneous air-flow measurements.[1,2,6] This instrument consisted of a microscopic platinum wire, 10 microns in diameter, suspended across the diameter of a 31.6-mm tube. One end of the wire was attached to a fine spring. When air flowed through the instrument, the displacement of the wire was recorded photographically by a moving-film camera. The deflection of the wire was linear with air flow, and its inertia, lag, and frequency of vibration did not interfere with respiratory measurements.

13.3 INSPIRATORY AIR-FLOW DETERMINATIONS

Studies were made[1] on a large group of subjects under sedentary and working conditions with no inspiratory resistance and with 25, 50, 76, 102, 152, and 203 mm of resistance (resistances were in mm water at 85 liters per minute). Work rates of no external load, 179, 415, 622, and 830 kg-m per minute were employed. Measurements of inspiratory curves and of minute volume were made for each subject under each condition. The photographic records obtained on all subjects were analyzed for respiration rate, maximum inspiratory air flow, length of inspiratory and expiratory cycles, sustained flow (two-thirds of maximum flow), flow above 85 liters per minute, and rise to sustained flow.

The data obtained showed that the earlier constant-flow standards for rating and testing canisters were inadequate. Maximum flows obtained when subjects performed maximum exertion were several times greater than the 32 and 85 liters per minute constant-flow test rates for canister penetration and breakdown and for resistance rating. It was recommended, for tests of canister penetration and breakdown, that a device be employed which simulated human breathing in frequency, amplitude, and shape of the inspiratory curves, and such a device was constructed. Using this technique, acceptance tests of production lots of canister charcoal resulted in the rejection of considerable quantities which had previously been thought to be satisfactory. The technique also reversed the order of the life test results with certain standard war gases adsorbed on a typical charcoal.

Maximum and average inspiratory air flows depend upon the amount of work being performed and the inspiratory canister resistance. Both of these factors must be taken into consideration in rating and testing protective canisters. With the values presented, the maximum rate of inspiratory flow can be estimated from the volume breathed under a wide range of conditions.

An extension of the above study was made with strenuous work carried to exhaustion under tropical and normal temperatures to observe the effect of strenuous exercise and temperature on air-flow measurements. Measurements were made on subjects walking and running on a treadmill with and without field equipment. Inspiratory and expiratory flows were determined[4] on the same subjects riding a bicycle ergometer. The salient findings of this study are given below.

The highest maximum air flows, minute volumes, and respiration rates were produced by soldiers carrying full equipment when running at 5.6 mph and approaching exhaustion. The highest mean values found with 17 subjects were 203 liters per minute for maximum instantaneous inspiratory air flow, 82.5 liters for minute volume, and 47 respirations per minute. The highest individual maximum inspiratory air flows occurred in most cases during the three minutes just preceding exhaustion. Over one-half of these flows occurred in the last minute of running before exhaustion. About 20 per cent of the subjects had their highest individual maximum inspiratory air flows during recovery, immediately following cessation of work. During a single breath, volumes of air as high as 1.1 liters with a peak instantaneous flow rate above 276 liters per minute were recorded. There was no significant difference in maximum inspiratory air flows, minute volumes, respiration rates, and shapes of inspiratory curves obtained under tropical environmental conditions when compared with those for the same subjects under normal environmental conditions. The most significant effect of the tropical environment was that it limited the endurance of the subject. The mean duration of the experiments was reduced about 20 per cent by the tropical environment.

The effect of increasing the resistance to inspiration (from 15 to 72 mm of water at 85 liters per minute) when the expiratory resistance remained the same (selected M-8 valves, 23 to 25 mm of water at 119 liters per minute) was to reduce the respiration rate (4 to 6 per cent), the minute volume (13 per cent), and the maximum instantaneous air flow (15 to 19 per cent). The length of the inspiratory cycle and the proportion of the cycle represented by the rise to sustained flow were both prolonged by the increased resistance.

No significant effects of a total inspiratory resistance of 72 mm (at 85 liters per minute) on endurance, pulse rate, body temperature, or pulse recovery were observed. It was evident from the reported subjective reactions that the above resistance was within the permissible limit of inspiratory resistance. Only when the amount of work performed was at the upper limit of the subject's working capacity did this inspiratory resistance perceptibly affect the subject's ability to complete the work.

One of the most important factors that affect the length of time to attain high maximum air flows, minute volumes, and respiration rates was the weight of equipment carried by the subject. The endurance of the subjects in these experiments was approximately halved when full equipment was carried. If the work performed was continued to exhaustion, maximum air flows of the same order of magnitude were obtained whether the subject was or was not carrying equipment.

The effort required during walking on the treadmill at 4.3 mph with full equipment resulted in inspiratory air flows equivalent to those obtained with a work rate of 1,107 kg-m per min on the bicycle ergometer. Running at 5.6 mph under the same conditions of resistance resulted in air flows, minute volumes, and respiration rates exceeding those in the bicycle ergometer work by 25 per cent for maximum flow and 40 per cent for minute volume and respiration rate.

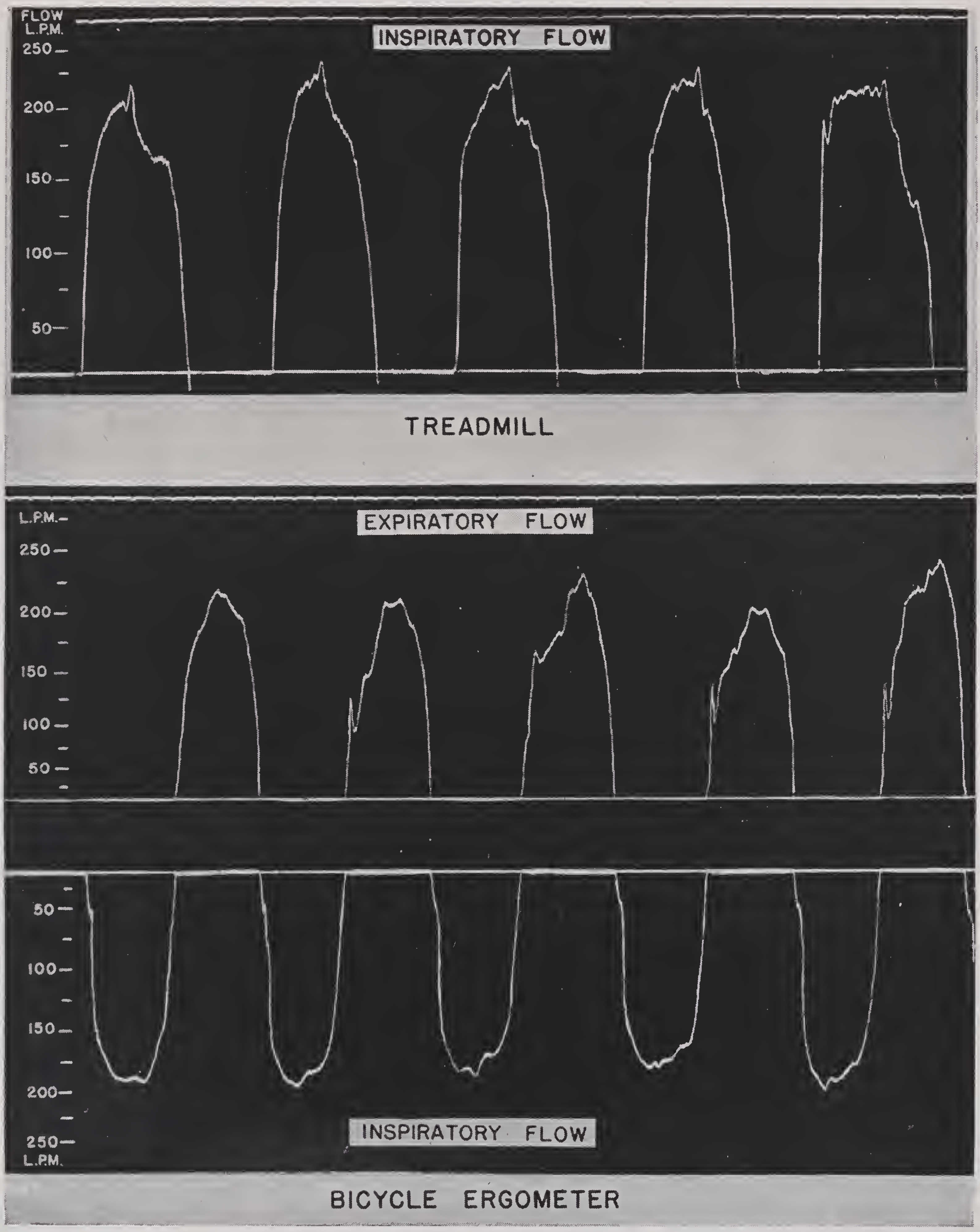

FIGURE 1. Respiration curves for men operating a treadmill and bicycle ergometer.

The effects of simultaneous inspiratory and expiratory resistance were to reduce the inspiratory and expiratory air flows, to reduce the minute volume and respiration rate, and to prolong the phase of the cycle in which the resistance was greatest. Usually the inspiratory cycle was prolonged. For all practical purposes, when both resistances are present, the expiratory cycle may be considered to be about 10 per cent greater than the inspiratory cycle.

A typical set of air-flow curves is shown in the accompanying Figure 1. These illustrations are reproduced from actual records taken during the experiments. The curves run from left to right. Time intervals are $1/5$ sec (indicated by notches in horizontal line at top). The treadmill curves were taken on a subject walking with full equipment at 4.3 mph and breathing through a combat service mask. The bicycle ergometer curves are for a subject riding at 1,107 kg-m (8,000 ft-lb) per minute and breathing through resistances comparable at maximum work.

13.4 CHARACTERISTICS OF INSPIRATORY AND EXPIRATORY VALVES

In order to determine the characteristics and nature of respiratory resistance of protective respiratory equipment, a study was made of all existing physiological, industrial, and service-mask valves.[2]

A study of 37 valves showed that the most important mechanical characteristics of these valves are their resistance to air flow, leakage, and opening pressure. All these factors were found to be related. Other important considerations are the location of the valve in the mask and the protection of the valve from damage and climatic conditions.

Comparison of various methods of determining the resistances of these valves to expiratory air flows indicated that the chamber test closely simulated the results obtained in tests with human subjects while sedentary or working. In the human tests, the air flow was measured by a modified instrument of the displaced wire type.

A detailed study was made of the resistance of the valves to various air flows, in chamber tests, and the data were plotted. The factors regulating the resistance to flow for various types of valves were analyzed. In these chamber tests, the curves of the resistances of the valves to increasing air flows, up to 250 liters per minute, were linear or nearly linear in many cases. To simulate the characteristics of valves in physiological respiration studies, a device having a linear relationship of resistance to air flow is recommended.

Tests of United States and foreign service masks showed that the lowest resistance to expiratory flow was offered by the Japanese army service mask. Modifications of the Japanese expiratory valve were developed which offered even less resistance. At a flow rate of 119 liters per minute, a resistance of less than 10 mm of water was obtained for a large-sized valve, and a resistance of less than 15 mm for a smaller valve. Both valves met the leakage specifications of the Chemical Warfare Service.

A new method of determining valve leakage, which corresponds closely to actual conditions in human respiration, was developed. The results obtained with this new method were compared with determinations of the static leakage of the valves when dry and when wet. Tests of leakage of an expiratory valve most representative of the conditions obtained during human use should be made when the valve is wet. The most practical method of expressing the valve leakage in these tests, to compare with the leakage under conditions of human use, was found to be the leakage per respiration.

Three methods of determining opening pressure were applied to expiratory valves, and their results were compared. Opening pressures that compared favorably with actual pressures of the valves in human use were those obtained when the valves were wet. Determinations of opening pressure, to be of significance, should be made with wetted valves.

13.5 EVALUATION OF MAXIMUM ALLOWABLE RESPIRATORY RESISTANCE

The object of this phase of the project was to determine maximum permissible amounts of inspiratory and expiratory resistance to breathing under conditions of heavy and medium work. The shape of expiratory air-flow curves and the relationship of inspiratory and expiratory air flows with varying amounts of resistance were observed. In addition, a study of the effect of physical condition on respiratory performance data was made.

The following conclusions were made on a basis of the data and observations obtained under heavy and medium work rates:

Inspiratory resistances up to 106 mm of water at 85 liters per minute and expiratory resistances up to 76 mm of water at 85 liters per minute separately do not interfere with the performance or completion of 15 minutes of heavy work (830 kg-m or 6,000 ft-lb per minute). A combined resistance of 106 mm inspiratory and 41 mm expiratory (total 147 mm) similarly does not hinder performance or completion of the above amount of work.

Physiological and subjective reactions indicated that for the heavy work rate (830 kg-m) resistances exceeding 82 mm inspiratory and 53 mm expiratory are not well tolerated separately or combined, although the work can be done. It is important to note that these values apply to subjects in fair condition without training for breathing through resistances. With training both for the work rate and for the resistance, it is probable that these values could be exceeded.

At maximal work, resistance becomes a more important factor. Subjects in good condition who can perform 1,107 kg-m (8,000 ft-lb) per minute of work for 15 minutes with minimal resistance (6 mm inspiratory and 3 mm expiratory) can breathe through resistances of 64 mm inspiratory and 41 mm expiratory and still perform the work without difficulty. Subjective responses indicate, however, that resistances above these amounts would not be well tolerated.

Inspiratory resistance alone above 64 mm does not cause so many subjective complaints as expiratory resistance alone above 54 mm of water. When expiratory resistance alone is present, or is greater than inspiratory at heavy work rates, oxygen consumption is lowered and oxygen debt may result. The addition of inspiratory resistance to such a condition increases the oxygen consumption to a value compatible with the work rate. Expiratory resistance should never exceed inspiratory unless both values are minimal.

Additions or reductions of 10 to 20 mm of water to either inspiratory or expiratory resistance will not produce any significant change in physiological or subjective reactions at heavy or medium work rates as long as the resistance is maintained within the limits stated above.

The results obtained at a medium work rate (415 kg-m or 3,000 ft-lb per minute) confirm the observations made at heavier work rates with the exception that higher resistance can be tolerated at the lower rate. This work rate can be maintained for over eight hours.

Individual variations were considerable for each resistance condition. The majority of the subjects, however, showed a decreased minute volume and a decreased respiration rate as respiratory resistance was increased. To compensate for these reductions, the oxygen deficit was increased, thus maintaining the required oxygen consumption for the work rate. The physiological mechanisms can apparently compensate for large changes in resistance by adjusting respiration rates, shapes of air-flow curves, and gas exchange values to maintain oxygen consumption.

Subjective reactions did not show correlation with objective measurements at resistance values of 64 mm of water at 85 liters per minute inspiratory and 41 mm of water at 85 liters per minute expiratory, which were comparable or somewhat greater than for the current service equipment. When the resistance is increased above these values, the number of complaints evoked was in proportion to the amount of inspiratory or expiratory resistance. Subjective responses are not a satisfactory criterion of physiological reactions to resistance unless the amounts of resistance exceed the limits stated previously.

Mean respiratory work rates formed only a small percentage of the total external work even when resistances of 106 mm inspiratory and 41 mm expiratory were present and a high percentage of subjective complaints were evoked. Respiratory work rate expressed as a percentage of the total external work may form a convenient basis for expressing permissible limits of resistance to respiration, since flow, resistance, and the external work rate are included. This study indicated that 1.2 per cent, corresponding, at a heavy work rate (830 kg-m), to 82 mm inspiratory and 53 mm expiratory resistances, is a reasonable limit of respiratory work which will not cause an appreciable number of objections to breathing through the resistance. Thus the allowable resistance for a given work rate can be determined from the minute volume, per cent of cycle values, and external work rate.

Observations on the subjects performing the experiments indicated that a practical rather than a physiological limit should be used for expiratory valve resistance in gas masks, since the resistance at which the face piece tends to "valve off" is below those found physiologically undesirable. The expiratory resistance should be low enough to prevent valving off when the suspension is adjusted so that the face piece fit is leakproof but the suspension tension is not so tight as to be uncomfortable. The lower the amount of expiratory resistance, the more readily this condition is attained.

Good physical condition resulted in superior performance of heavy work. A comparison of athletes and non-athletes of the same age group, each performing bicycle ergometer work without previous ergometer experience, showed the athletes to be much better than the non-athletes. The outstanding differences shown by the athletes were a lower working pulse rate, a lower minute volume, lower maximum flows, decreased oxygen consumption, and an increased oxygen deficit.

Test-retest values correlated well indicating that there was not any significant training effect in these experiments. The coefficients had high values, which checks the reliability of the measurements. Good correlations were found between oxygen consumption and pulse rate. The correlation of these two values was reduced significantly when expiratory resistance was present, possibly indicating some effect of expiratory resistance on the circulation.

The mean data obtained indicated that oxygen deficit, respiration rate, and minute-volume changes are a function of total resistance. As the total resistance (inspiratory plus expiratory) increased, oxygen deficit increased, and respiration rate and minute volume both decreased. When the resistances were above minimal values, the maximum air flows, air-flow curve proportions and shapes, and flow ratios were in proportion to the ratio of inspiratory to expiratory resistance. Pulse rate changes were not significantly altered by resistance. They were, however, affected materially by physical condition and adaptation to work.

Protective respiratory equipment employing resistances which exceed the limits found in this study should be modified so as to reduce their resistance within the desirable limits. Suggested maximum limits are 82 mm of water at 85 liters per minute for inspiration and 53 mm of water at 85 liters per minute for expiration.

Expiratory resistance should never exceed inspiratory in any protective equipment unless both amounts are minimal. Rebreathing devices should be designed so that they do not require exhalation through resistance (carbon dioxide removal canister), but rather that the air to be purified should be drawn through the canister during inspiration.

The effect of training on performance of subjects breathing through resistance deserves further investigation. General physical training will reduce air-flow requirements for a given work rate considerably (20 per cent). It is probable that training with resistance will decrease air-flow requirements and also permit higher resistances to be used for greater protection.

The data presented on the effect of inspiratory and expiratory resistance on the shape of the air-flow curves and maximum air flows can be applied to the design of simulated breathing devices and valves for protective equipment.

The question of subjective reactions to resistance was studied in a short investigation.[3] Service masks with the canisters mounted on the face pieces (combat) and service masks with canisters in carriers were studied. Soldiers had reported that service masks with the canisters mounted on the face pieces had lower resistance, but at steady test air flows this difference did not exist. Measurements were made with static (steady) and dynamic (simulated breathing) air flows to see whether the reported differences were due to dynamic air movements. No difference was found.

13.6 FIELD STUDIES

In conjunction with the work on inspiratory air-flow determination, a portable air-flow measuring instrument was developed.[5] By means of this instrument it was possible to make air-flow measurements of soldiers performing various field operations. A preliminary study of coast artillery, obstacle course, and tank operations was made.[7] The data obtained on field studies are of value in estimating requirements and life of protective respiratory equipment.

13.7 FUTURE WORK

The measurements of inspiratory flow and their magnitude indicate that some development might be directed towards use of devices to mechanically reduce the peak flow of air. It was also observed that training and physical condition have an important effect upon the volume of air breathed and the maximum air flows which result when moderate to exhausting work is performed. Further study on the effects of training on air flow and tolerance to resistance is needed. The study made on this project indicates that subjective reaction to resistance can be materially reduced by training or gradual acclimation to increasing amounts of resistance.

The study of air-flow requirements for various field operations should be continued in order to determine the amount of protection needed and the life of the protective device.

Chapter 14

SABOTAGE OF GASOLINE ENGINES

Because of the extensive mechanization of warfare, there is a continual requirement for effective methods of causing gasoline engines to become inoperative, either in order to immobilize combat vehicles or to disrupt movement of supplies.

14.1 PROKNOCKS

Since the discovery made during World War I that small amounts of certain compounds added to gasoline, or admitted through the carburetor air stream, would make a motor knock with resulting loss of power, many suggestions were made that such materials be put to military use as a means of impeding enemy activity. These suggestions have ranged from creating gas clouds in front of attacking airplanes to adding a proknock to gasoline for sabotage purposes. Outstanding among these suggestions was the proposed use of the latter agent for immobilizing planes at the time of take-off from enemy fields.

This investigation was started by measuring the effectiveness of all varieties of available compounds, with particular reference to those previously reported as showing promise. All compounds showing even slight activity were found to contain one or more of the following elements: nitrogen, phosphorus, arsenic, antimony, oxygen, sulfur, chlorine, bromine, and mercury. It further became apparent that compounds consisting exclusively of these elements showed, in general, stronger action than compounds containing other elements in addition.

All tests were made in a standard CFR knock test engine using the American Society of Testing Materials method (ASTM D 357-40). The principal fuels used were non-leaded base for 100-octane aviation gasoline and the same base treated with approximately 2.8-cc tetraethyl lead in ethyl aviation fluid. In some tests the materials were added to blends of benzol and a straight run gasoline of low octane number treated with several concentrations of lead.

Approximately 250 materials were tested in varying concentrations with one or more of these fuels.[2] The materials were introduced into the engine in solution in the fuel, in a solvent in solution in the fuel, in solution in a solvent introduced into the air intake system, as solids delivered at the intake valve, or as a vapor or gas introduced into the air intake system. In most cases the method used was dependent upon the characteristics of the material being tested, though some comparisons of methods were made.

Many of the materials tested had little or no effect on the antiknock value of either untreated fuels or fuels treated with tetraethyl lead. Some materials greatly reduced the octane number of both leaded and non-leaded gasolines, while others reduced the octane number of leaded gasolines only. A few materials reduced the antiknock value of untreated fuel without appreciably changing the value of leaded gasoline, and in a few instances materials were knock suppressors in the untreated fuel but induced knock in the treated gasoline. Typical experimental data are shown in Figure 1.

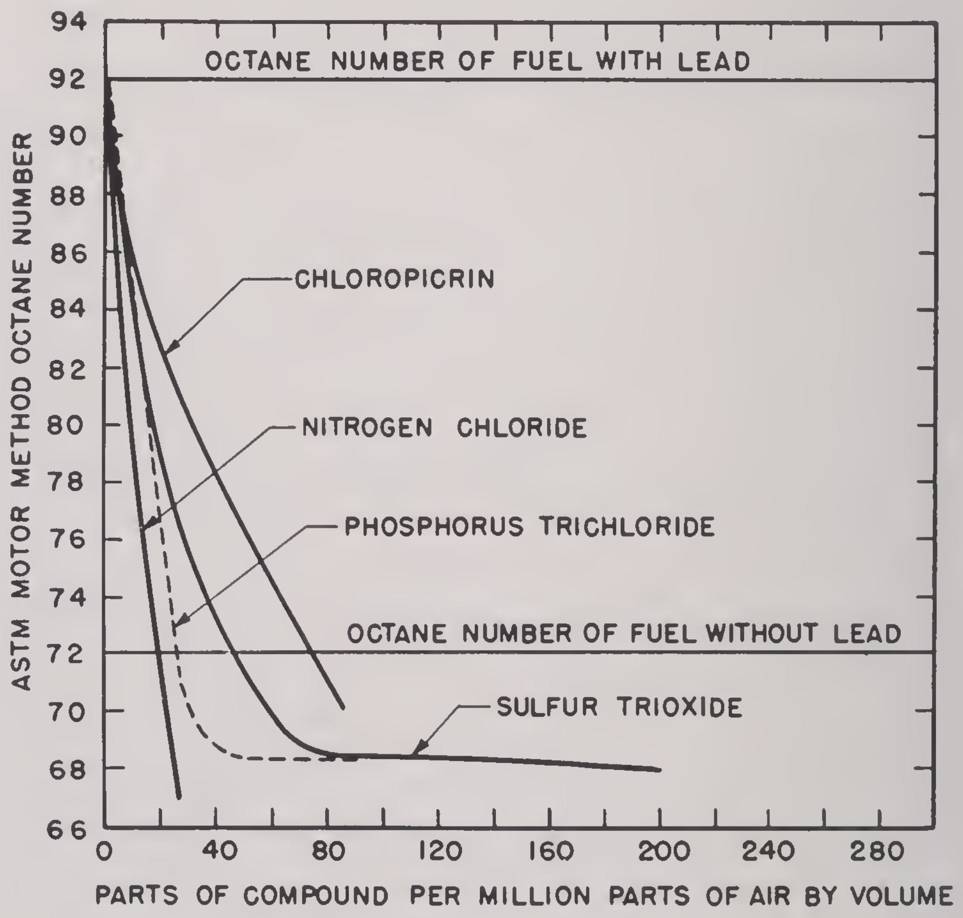

Figure 1. Test results of proknock materials.

The most effective materials found are listed below. The concentration given is the amount of material required to reduce the antiknock value of the fuel by 12-octane units.

	Concentration, parts of material per million parts of air to effect a reduction of twelve in octane number	
Material	By weight	By volume
In Lead-Treated Fuel		
White phosphorus	6.4	...
Nitrogen chloride	29	7
n-Butyl dichlorophosphine	44	8
Arsenic trichloride	49	8
Arsenious acid anhydride	50	8
Sulfur trioxide	60	22
Sulfur dichloride	62	18
Sulfur monochloride	68	15
1,1-Dichloro-1-nitroethane	72	15
1-Chloro-1-nitropropane	80	19
Phosphorus sulfochloride	85	15
Phosphorus trichloride	87	19
Ethyl thionitrite	88	29
FS (55% sulfur trioxide, 45% chlorosulfonic acid)	90	...
Nitrosyl chloride	95	42
In Non-Leaded Fuel		
Nitrogen chloride	148	36
2-Ethylhexyl nitrite and chloropicrin*	270	48
2-Ethylhexyl nitrite	290	53
Chloropicrin and isoamyl nitrite*	317	62
2-Chloroethyl nitrite	365	97
Isoamyl nitrite	500	123
Chloroform and isoamyl nitrite*	550	134
Isoamyl nitrite and *n*-butyl sulfide*	550	122
Amyl nitrate	620	135
Chloropicrin, isoamyl nitrite, and *n*-butyl sulfide*	620	121
Isoamyl nitrate and *n*-butyl sulfide*	680	141
Isoamyl nitrate	700	153
n-Butyl sulfide and chloropicrin*	720	132
Tertiary butyl thionitrite	750	182

* Equal parts of each component.

The effectiveness of any of these materials will depend on how they are used. For overall value, nitrogen chloride proved itself the strongest proknock. All other agents tested varied their relative effectiveness with the type of fuel and their concentrations. Hence to select the most suitable substitute for nitrogen chloride, it is necessary to decide in advance the optimum concentration and to know the type of fuel being used by the enemy. Nitrogen chloride has the disadvantage of being difficult to handle because of its instability. It decomposes slowly even at the freezing point, and it explodes at 201 F or on contact with some organic materials.

The obvious disadvantages of the use of a proknock material for dispersal on enemy air fields to prevent take-off of planes are:

1. The quantities required to create effective gas clouds are excessive. (The concentration of 29 ppm by weight required to reduce the octane rating 12 units corresponds to 300 pounds of agent uniformly dispersed in the air to a depth of 50 ft over an area of 1 square mile.) It was decided that the same weight of high explosive would be more effective.
2. Air movements of even moderate proportions will rapidly dissipate gas clouds and hence cause the effectiveness to be only transitory, requiring no effort on the part of the enemy for its removal.
3. Any effectiveness disappears at part throttle, or low barometric operation of motorized equipment. This must be taken into account when considering warfare against tanks and aircraft at high altitudes. No data were obtained on whether the octane number reduction which could be obtained was sufficient to prevent take-off of planes.

Tests were made to determine the effect on a Plymouth engine of reducing the octane number of the fuel. The engine was adjusted for a trace knock at 1,200 rpm and full throttle using 70-octane fuel. Other fuels were blended to give 60-, 50-, and 40-octane number and were substituted for the 70-octane fuel. The 60- and 50-octane fuels caused knocking, but did no damage to the engine in 5 minutes of operation. Forty-octane fuel caused extremely heavy knocking. The engine was operated for 20 minutes, which is far longer than an engine in such distress would be driven under ordinary circumstances. No damage was done and the observed blow-by was constant during this time. These tests indicate the futility of trying to destroy an automotive engine by lowering the octane rating of the fuel.

14.2 GASOLINE SABOTAGE[8]

At the time of the German drive through Holland, Belgium, and France in 1940, large quantities of gasoline were abandoned and used by the Germans. Because of storage conditions, particularly underground storage, it was very difficult to sabotage these supplies with explosives or incendiaries, and a request was made for a contaminant which could be added to the fuel to make it unfit for use. As the Allies changed from the defensive to the offensive, the requirement changed from sabotage of "friendly" gasoline to that of "enemy" gasoline. The use visualized also changed from sabotage of bulk supplies to sabotage of drum lots, or the fuel of individual vehicles. These changes, however, did not appreciably alter the desired properties of the material sought. The specifications for the ideal material were that it be nondetectable and a small quantity be required, that it ruin any engine, that it mix with the fuel automatically, and that it work in all types of gasoline.

The best material found was an oil-soluble phenolic resin made by condensing a para-tertiary alkyphenol with formaldehyde. This product is sold by the Paramet Chemical Corporation and is known as Paradura 10-P. It is a solid and heavier than gasoline but readily soluble, and is not easily detectable after solution. It should be prepared as fine aerated granules which will float on gasoline. It can be used as a 50 per cent solution in gasoline, but this increases the weight of material to handle. The Paradura was deposited on the valves and valve stems of a running motor, causing sticking of the valves. When one-half gallon of fuel containing 20 grams per gallon was passed through each cylinder of a motor, the intake valves began to stick open and the motor missed badly. When the motor was stopped, it could not be restarted. Continued use resulted in stalling of the motor.

Other effective compounds in the order of their effectiveness were:

1. Soluble phenolic resins.
    a. Paradura No. 367.
    b. Super Beckacite No. 1001.
2. Rosin ester (ester gum), or Santoresin.
3. Chinawood oil.
4. Phosphorus trichloride or phosphorus dichloronitride ($PNCl_2$).

Twenty grams per gallon is an effective concentration for these materials. Ten grams can be used, but twice or more the running time is necessary. The phosphorus compounds caused sticking of the piston rings; the others primarily caused valve sticking.

Over 225 compounds were evaluated in one-cylinder air-cooled Delco motor-generator units, using 1 quart of treated gasoline for each run. The materials showing any appreciable effect were given longer tests, using several gallons of fuel. The best of these were then checked in block tests with a multi-cylinder motor, using 6 gallons of fuel for each run, and a final check was made by a road test using a 1½-ton Ford truck.

14.3 SABOTAGE OF LUBRICATING OIL

Methods of sabotaging motor vehicles by additives to the lubricating oil were investigated in the hope of finding a material which was more effective than the fuel additives. Several materials were tested which caused severe sticking of the engines. The reaction product of *m*-benzenedisulfonic acid and mesityl oxide, when added in concentrations of 1 to 2 per cent by volume to lubricating oils of the high viscosity index, nonadditive type, caused engines to stick so tightly from varnish as to be inoperative.[10] In concentrations of 2 to 4 per cent it was effective also in oils containing additives of the detergent type. When this material was poured directly into the crankcase of an engine, it caused the engine to seize tight after being driven only a few miles.

The mesityl compound was prepared by dissolving, with stirring, 100 grams of *m*-benzenedisulfonic acid in 150 cc of mesityl oxide. Cooling was required to keep the temperature at 65 to 70 C, which should not be exceeded. This reaction product is very viscous and difficult to dissolve in lubricating oil; before use it should be thinned with 40 per cent acetone. A slow reaction with the acetone occurs, which increases the viscosity so that the thinning should be done within a few days of intended use.

Other agents found effective were:

Reaction product of acetone and benzenedisulfonic acid.

Benzenedisulfonic acid and aldol (added separately).

Benzenedisulfonic acid and crotonaldehyde (added separately).

Furfuryl alcohol.

A compound labeled RX furnished by Tidewater Oil Company (composition unknown).

A mixture of the following composition:[9]

Phosphorus	14.7 per cent
Linseed oil acids	63.5 " "
Rubber	2.5 " "
Ethyl iodide	3.5 " "
Carbon disulfide	15.7 " "

Additional work on sabotage of fuels and lubricating oils was carried out at the request of Division 19. The results are reported in the History of Division 19, dated June 30, 1945.

Chapter 15

PRODUCTION OF POTABLE WATER FROM SEA WATER

15.1 INTRODUCTION

Drinking water is essential for all military operations and often the only available source is sea water. Various types of distillers had been used for shipboard use but these were too cumbersome and hazardous to use on life rafts. Only a few days supply of water could be carried on the inflatable rafts used by aviators forced down on the ocean, and often this was not sufficient for the men to survive until rescued. An investigation was, therefore, undertaken for the development of methods of obtaining potable water from sea water either by distillation or by chemical methods. Chemical and electrolytic methods for purification of water were also investigated as of possible application to shipboard use in preference to distillation.

15.2 ELECTROLYTIC METHOD

The electrolytic method depended on attracting cations and anions toward electrodes separated from the water to be purified by permeable membranes. This method had been found impractical mainly because of the ohmic resistance of ordinary fresh water. However, it seemed possible that a combination of new reagents with electrolysis might be worked out for sea water. The plan was to partially purify water by the ordinary electrolytic method and then recover the acid and base formed in the electrode compartments for use in regenerating exchange materials to be used for the removal of the remaining solids in the water. The electrolysis operation was not satisfactory, however, because of back diffusion through the membranes. Only about 1 per cent reduction of total solids was accomplished.

15.3 CHEMICAL METHODS[1,2]

All the commercially available base exchange materials were investigated for their effectiveness in removing salts from sea water. These included Zeo Karb H, Catex, Amberlite IR-1, Alkalex, Deacidite, Anex, and Amberlite IR-4. Base exchange materials are solid substances which react with ions in solution, with the resultant removal of the ions to the solid substances, the ion being replaced in solution by another ion originally on the solid. The most efficient cation exchanger found was Amberlite IR-1 supplied by Resinous Products Company. This material had a capacity of 1.36 milliequivalents per gram. The best anion exchanger was Amberlite IR-4 from the same producer, which had a capacity of 4.7 milliequivalents per gram. After these materials are exhausted the cation exchanger can be regenerated with sodium hydroxide, the anion exchanger with sulfuric acid. The regenerating chemical can be dissolved in sea water and the sea water used for washing the resin. Using the sea water for regeneration reduces the efficiency of the exchanger, but this was taken into account in the figures given above. It was found possible to produce water of essentially zero mineral content by the use of these resinous exchange materials. The necessary filter beds and other equipment for a shipboard installation would weigh about 1 pound per gallon per day. This is the same as the installed weight of the standard double effect evaporators. However, for 1 gallon of water obtained, the regeneration of the exchange resins would require 1.25 pounds sulfuric acid for the removal of the cations and 0.4 pound sodium hydroxide for the removal of the anions. The total weight of chemical necessary, therefore, for the purification of sea water using the exchange resins is about 1.65 pounds per gallon as compared with 0.35 pound fuel oil per gallon of distilled water required by the double effect evaporators. It was concluded that the chemical methods studied could not compete with the double effect evaporators for shipboard operation.

The use of heavy metal oxides for the removal of chlorides from sea water was also investigated. Mercurous oxide removes chloride from solutions and the precipitated mercurous chloride can be converted to the oxide with sodium hydroxide. The reaction of the mercurous oxide with chloride was practically complete; when the theoretical amount of oxide was added, only a trace of chloride and mercury were left in solution. This reaction took place only in an acid solution, however, and was of no value in neutral sea water. The use of exchange resins to produce acidity in the sea water before treatment with mercurous oxide was unsuccessful. Silver oxide precipitated the chloride in a neutral solution, but an excess of the reagent was required and the reaction was slower than with mercurous oxide. Lead oxide removed only about 25 per cent of the chlorides. None of these reactions were considered satisfactory for use.

Although the exchange resins were not considered satisfactory for shipboard use, several modifications were developed which might be of value on life rafts where regeneration of the chemical is not desired. The best of these involved the use of barium to precipitate the sulfate. Because of the toxicity of barium, it is desirable to use less than the theoretical amount for precipitation of all of the sulfate. This would insure no barium being left in the water, and the small amount of sulfate remaining would not be harmful. The solution was then filtered and treated with a slight excess of Amberlite IR-1, which had been previously treated with silver nitrate. The cations were taken up by the exchange material and the silver ions freed to precipitate the chloride from the sea water as insoluble silver chloride. The solution was then filtered yielding a water with a total mineral content of 200 ppm. The ratio of water formed to materials used was 6 to 1 by weight. Thus, six times as much drinking water could be obtained by using this chemical as could be carried.

15.4 SOLAR STILLS[4]

The amount of solar energy received on a horizontal surface on a clear summer day, or average tropical day, averages at least 2,000 Btu per sq ft. In a simple distillation process operating at atmospheric pressure, about 1,100 Btu are required per pound of distilled water. Therefore, if the solar energy could be utilized completely for simple distillation, the average distilled water obtained would be 1.8 pounds of water per sq ft per day. Numerous attempts have been made to use solar energy for distillation. The basis of all solar distillers consists of a boiler or evaporator which is a blackened pan, or receptacle, containing the feed water. Since the evaporator serves the purpose of absorbing solar radiation, it should present an extended black surface which does not fade in color under operating conditions. Its heat capacity should be small in comparison with the heat capacity of the feed water for one day's operation. The condenser consists of an inclined surface which transmits solar radiation as fully as possible. The condensing surface may advantageously surround the evaporator on all sides terminating in a collective device to accumulate the distillate. In operation the absorbed solar radiation heats the feed water held in the evaporator. Water vapor is generated which in turn condenses on the inner surfaces of the condenser and is collected by suitable means. The process obviously occurs at temperatures well below the boiling point of water and depends on the diffusion of water vapor, through air, from the evaporator to the condenser. The condenser surface is inclined to facilitate the flow of the condensate.

Certain losses in the operation of a solar distiller are unavoidable. Clear glass and most transparent plastics suitable for use as the condenser absorb a few per cent of the incident radiation, and about 8 per cent is lost from reflectivity, giving a total loss of about 10 per cent. Film-type water condensation on the inside of the surface does not lower the transmission of the radiation and is preferable to dropwise condensation. Heat losses by reflection at the evaporator-absorber surface may be caused by imperfect blackness. Surfaces showing a flat black finish absorb about 96 per cent of the incident solar radiation. The surface of the absorber emits long infrared radiation corresponding to its temperature. Calculation shows that this heat loss may amount to about 12 per cent of the original figure. Heat loss from the evaporator absorber due to dry air circulation is relatively small, generally not more than 5 per cent of the incident energy for most arrangements of the surfaces. The heat loss from the bottom side of the evaporator constitutes the most variable factor. This loss was a major cause of the low efficiency of early solar distillers. The trays containing the feed water were made of wood painted black and generally placed upon the ground. The thermal conductivity of wood is comparatively high and a relatively large amount of solar heat was wasted in heating the ground. Placing the tray upon thermal insulation equivalent to about 1 in. of insulating board diminishes the bottom heat loss to about 10 per cent of the initially incident energy. Instead of the insulating board, its insulating equivalent of other materials, or layer-built structures with air spaces, may be used and would be preferred from the point of view of compactness. The above losses indicate that this type of solar distiller should show a maximum efficiency near 60 per cent.

For use on life rafts compactness of the packed still is esssential. In order to accomplish this compactness, a still was designed which was made entirely of flexible, foldable materials. This enabled the still to be folded into a very compact package but when opened up it could be inflated to retain the desired shape and size for the practical production of drinking water from sea water. The outer envelope of the still designed was made from a transparent vinylite plastic, which, when inflated, had a circular pillow shape. The inner surface of this plastic envelope served as the condensing surface for the water vapor. On the bottom side the envelope was equipped with a water-collecting bag into which the distilled water drained. An outlet was attached to the water bag which could be closed with a plug. Inside the plastic envelope a black absorbent pad was held in a horizontal position by waterproofed fabric strips. This pad was made from a regenerated cellulose sponge containing a permanent black dye.

For operation, the folded still was removed from its container and unfolded. A collapsible funnel, which was part of the equipment, was inserted into the outlet and the still filled with sea water through the funnel, to saturate the black absorbent pad. The funnel was removed and the device inflated through the rubber outlet, either by mouth, or by means of an air pump. The inflated still was closed with the plug and the device turned into position with the water-collecting bag on the bottom. The excess sea water was permitted to drain for about 10 minutes, collecting in the water bag from which it was removed by temporarily opening the plug. The device was then ready for operation. It was designed to float in the water beside the raft, needing no attention except for the removal of the drinking water from time to time. When the device was exposed to the sun, most of the solar radiation was transmitted by the transparent envelope and absorbed by the black absorbent pad, heating the sea water with which the absorbent pad was saturated. Water vapor was generated, condensing on the inner surfaces of the envelope, which was cooled by the air on the upper surface and by the water on the lower surface. The condensed distilled water trickled down the inner side, collecting in the water bag. A slit opening above the center of the water bag acted as a valve and prevented the collected distilled water from being splashed back into the distilling compartment.

The final model[a] which was designed had a black absorbent pad of 2-sq ft area. When exposed to bright sunshine, the still produced about 4 oz of water per hour with a probable tropical daily yield of 1 quart of drinking water. The folded volume of this model was about 70 cu in. (1.2 quarts), including the folding funnel and the carrying bag, and its weight was about 1 pound.

The inner side of the transparent envelope should be coated with an agent which produces film-type condensation of the water vapor. Dropwise condensation absorbs part of the energy striking the surface, which results in less heat being transmitted to the absorbing pad and also causes some revaporization of the condensed water, resulting in a lower yield. A good anti-fogging coating was obtained by spraying or brushing over the vinylite film a solution containing 15 per cent, by weight, of polyvinyl alcohol dissolved in a solvent of equal volumes of water and alcohol. To produce better adhesion, the solution was mixed with about 10 per cent of acetone or similar solvents, capable of leaving vinylite slightly tacky. After drying, the sheet was baked at 180 to 200 F for a short time. Coatings of this kind were used in continuous operation for two months without showing any deterioration.

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[a] The basic development of the still described above was completed by the contractor prior to the initiation of the OSRD contract. OSRD suggested financing the manufacture of a number of prototype models for tests by the Services.

In tests run off the shore of Miami, Florida, these stills operated at an efficiency of 49 per cent. Figure 1, taken at the time of the Miami tests, shows two inflated stills being operated from a raft. Stills of this type were adopted by the Army as part of the life raft equipment.

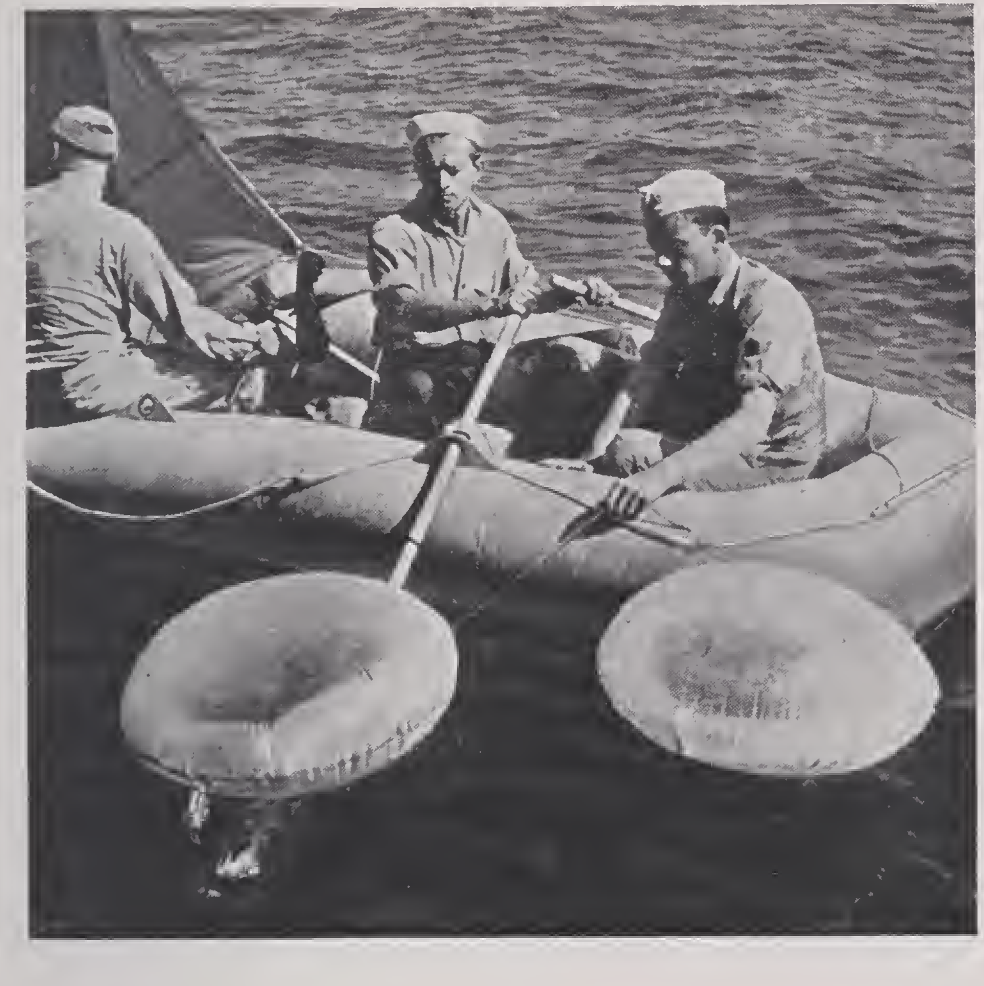

FIGURE 1. Two solar stills being operated from a life raft.

15.5 HAND-OPERATED VAPOR COMPRESSION STILL[3]

The vapor compression type of still has been used with satisfactory results by the Navy for shipboard use and by the Army for land use. This type of unit has a very high thermal efficiency, achieved through continual re-use of the heat required for vaporization of the sea water by efficient heat exchange between incoming and outgoing streams. A compressor compresses all the steam from the boiling section of the evaporator and discharges it at an increase of 1½ to 3 psi to the condensing section of the condenser. This pressure differential serves to raise the condensation temperature of the steam in the condenser sufficiently above the boiling point of the water in the evaporator, so that the heat of condensation will be transferred to the boiling sea water and thereby supply the heat of vaporization for the latter. If it were not for loss of heat to the surroundings and loss of heat in the streams of product and concentrated overflow, only a very small amount of power would be required for operation of the steam compressor. Even with these heat losses the ratio between product output and energy input is quite large; the actual energy input as work is of the order of 5 per cent of the latent heat of evaporation.

Because of the high efficiency of this type of unit, a model was designed and built which utilized the energy input from manual operation, and which was small enough and light enough to be carried on lifeboats and possibly on the large inflatable life rafts. The still developed was 10 in. in diameter and 12 in. high and weighed 17½ pounds without fuel. It produced distilled water at the rate of 1 pound per hour when supplementing the mechanical energy input with a small amount of heat. With mechanical input alone, the production was reduced to about ⅔ pound per hour. The starting time was 1 hour when the mechanical input was supplemented with heat and was about 2 hours when no supplementary heat was used. By use of heat alone the unit was brought to operating temperature in about 1½ hours. The amount of manual effort required for production of a pound of water was very much less when supplementary heat was used. When using supplementary fuel, the preferred rate of fuel consumption was found to be of the order of 1 pound for each 57 pounds of water produced (based on the use of methyl alcohol as fuel). The mechanical energy was supplied by means of a manually operated crank at the top of the unit.

Chapter 16

UNDERWATER COATINGS

16.1 SUMMARY

Complete coating systems for Navy vessels were investigated by means of laboratory studies, submerged panel tests, and trial applications to vessels of various sizes. Application of sand to partially dry primer was found to provide a good anchor for the topcoat, and to improve adherence of topcoat to primer. A wash of phosphoric acid and polyvinyl resins in a dilute alcoholic solution provided an excellent pretreatment of clean steel, and improved adherence of the primer. Both wet sand blasting and a special mechanical tool were found satisfactory in cleaning ships' hulls prior to painting.

An improved primer was developed containing polyvinyl butyral resin pigmented with zinc tetroxychromate. This was found markedly to reduce blistering. Blistering was found to be due to osmosis caused by increased hydroxyl ion concentration resulting from corrosion of the steel underneath coatings. Various parts of a ship's hull may be anodic and parts cathodic—corrosion occurs on the anodic areas and blistering where the surface is cathodic. Laboratory techniques were developed for studying potential changes of corroding areas, and for measurement of the electrical resistance of coatings. The latter were especially valuable in indicating tendencies to blister.

A great many antifouling coating formulations were investigated, and the mechanism of fouling protection studied in the laboratory. A technique of measuring the rate of leaching of the toxic copper constituent from the coating by sea water was developed, and it was established that fouling protection was obtained if the leaching rate was greater than 8.2 mg copper per 24 hours from 1,000 sq cm. The other principal variable affecting fouling protection was the extent and character of the insoluble green copper complex deposited on the surface after immersion in sea water. Improved antifouling coatings were developed containing cuprous oxide, rosin, and vinyl resins. These permitted the use of 4 to 5 parts pigment to 1 part binder, and gave excellent antifouling protection for more than 18 months. A new formulation employing flake copper in place of cuprous oxide found considerable application for wood hulls.

A study of paint removers showed that several formulations based on methylene chloride were effective, and were noninflammable as compared with the common acetone-benzene mixture.

Anaerobic sulfate-reducing bacteria were found to play an important role in marine corrosion, causing the formation of black iron sulfide next to the steel. Bacteria were also found to attack coatings, and a preliminary study indicated that certain paint constituents, notably paraffin, were selectively attacked.

A study of the fire hazard due to protective coatings on interior ship surfaces showed that the paint thickness should be kept at a minimum if the spread of fire from one bulkhead to another was to be prevented. Essentially complete protection was obtained by using not more than three coats of a paint containing not over 20 per cent binder, and by using antimony oxide as 10 to 20 per cent of the pigment. The Navy No. 84 primer was found to contribute to the flashing and burning; this was corrected by adding aluminum flake directly to the standard No. 84 primer before application.

16.2 INTRODUCTION

Coating systems for ship bottoms should consist of two components: the anticorrosive paint, applied directly to the cleaned metal, and the antifouling topcoat. The purpose of the anticorrosive paint is to protect the metal from corrosion and to act as a base for the antifouling coat. It is necessary that it have good adhesion over long periods of exposure to both the metal and the topcoat. Regardless of its other properties, failure in adhesion will result in fouling or corrosion or both. Other necessary properties are high moisture resistance, as measured both by permeability and by the softening or weakening of the film during prolonged contact with water; resistance to weak alkalies; the ability to form a tough film without excessive strains, shrinking, or expansion during drying; and the retention of its properties over the temperature range encountered from the tropics to the subpolar regions. The purpose of the antifouling paint is to prevent the attachment or growth of fouling organisms.

While the investigation of underwater coatings began with the emphasis on the antifouling coat, it was soon apparent that the best antifouling paint was worthless unless it could be held on for a reasonable length of time. A satisfactory primer was therefore essential to a satisfactory antifouling paint. Further, the primer was greatly affected by the surface over which it was applied. Hence, a successful paint system is equally dependent upon surface preparation, primer, and antifouling paint.

16.3 ADHESION, BLISTERING, AND CORROSION

16.3.1 Mechanical Improvement of Adhesion

Adhesion between the standard Navy primer and the hot plastic antifouling paint was not good and an attempt was made to correct this by mechanical methods, which would not necessitate changes in the formulation of the paints. It was believed that increasing the available surface to both the primer and the plastic would improve the apparent adhesion, and granulated slate, sand, fiber glass, and aloxite were evaluated for this purpose. It was found that considerable improvement in adhesion and in the tendency of the plastic to sag at elevated temperatures was obtained by embedding sand in the partially hardened primer. This produced a surface similar to sandpaper with the granules protruding well above the surface. After the primer had dried thoroughly the hot plastic was applied with a hot spray gun. Impact tests of the standard system caused failure from lack of adhesion of the plastic to the primer. With the sand granules, more drastic treatment was necessary to cause failure, and the failure finally occurred from lack of adhesion of the primer to the metal.

16.3.2 Primers and Corrosion

Uncoated steel corrodes quickly in sea water, eventually yielding either the common red oxide (Fe_2O_3) or the black oxide (Fe_3O_4), both in the hydrated form. These same reactions may occur underneath a paint film. The formation of iron oxides is accompanied by a considerable expansion in volume and the development of a weak spongy deposit which pushes the paint film away from the metal and eventually destroys its continuity.

These visible evidences of corrosion are accompanied by chemical changes which also exert a destructive action on some types of paint films. Metallic iron in contact with aerated sea water first passes into solution as ferrous iron, forming ferrous chloride and sodium hydroxide. The ferrous chloride may change quickly to ferrous hydroxide and then more slowly to the black iron oxide (Fe_3O_4, $4H_2O$), or the red iron oxide (Fe_2O_3, $3H_2O$), depending upon the amount of oxygen available. The alkalinity developed in contact with the underneath side of the paint film, due to the presence of sodium or magnesium hydroxides, is an important factor in the destruction of paint films in salt water. If the paint film is of a composition which may be saponified easily, it quickly softens and loses adhesion under this attack and permits corrosion to spread to adjacent areas. Thus, the use of vehicles having sufficient moisture and alkali resistance to withstand saponification is indicated. For instance, such materials as rosin or raw drying oils are easily saponified and have generally yielded poor results in primer vehicles, while several types of synthetic resins and heat-polymerized drying oils have much greater resistance to saponification and have yielded excellent results. Other synthetic products such as chlorinated rubber, polyvinyl chloride, and polystyrene also have excellent resistance to alkali and may prove useful in primers.

Corrosion usually begins at isolated points where some defect in the paint film or in the cleanliness of the surface at the time of application permits the above reactions to start. It is almost impossible to avoid such defects entirely; the practical control of corrosion depends largely upon preventing it from spreading beneath the film from these localized spots, which is largely a property of adhesion.

The chemical and physical factors that govern adhesion, especially for underwater coatings, are so numerous and complex that the determination of satisfactory performance has had to depend almost entirely upon empirical tests. However, the requirements for ensuring adequate adhesion to metal may be stated briefly as follows:

1. Ability to "wet" the metal surface completely in order to obtain initial adhesion and avoid bare or thin spots during application.
2. Presence of "polar" groups to ensure a permanent bond after evaporation of any volatile constituents.
3. High moisture resistance, as measured both by permeability and by the softening or weakening of the film in prolonged contact with water.
4. High resistance to weak alkalies to avoid the saponification and softening action of alkalies developed beneath the film.
5. Ability to form tough, strong films without setting up excessive strains by shrinkage or expansion during or after the drying process.
6. A sufficiently wide range of temperature stability to ensure that the coating will not become too soft or weak at 90 to 100 F, or too brittle at 20 to 30 F.

Unfortunately, no one bonding material now known possesses all these properties in a high degree; therefore it becomes necessary to formulate mixtures of the available materials to obtain the most suitable range of properties.

The use of phosphoric acid in dilute aqueous or alcoholic solutions has long been established as one of the best known methods for pretreating steel, zinc, and other metals to produce a passive surface and improve adhesion of subsequent coatings. For best results it has been necessary to use a hot dip process and careful control, which is impossible on ship hulls or other large structures. When phosphoric acid solutions are used cold, especially under variable field conditions, results are very erratic.

It was found that mixtures of phosphoric acid and polyvinyl resins, applied to clean metal surfaces in dilute alcoholic solution, produce exceptionally uniform, adhesive, and tough deposits to which subsequent paint coats tenaciously adhere. The superiority of this treatment over phosphoric acid alone, Deoxidine, and other proprietary products was most striking in the case of galvanized iron or sheet zinc, which have always been most difficult to paint satisfactorily.

A film composed of 20 to 35 per cent phosphoric acid and 80 to 65 per cent polyvinyl butyral resin pigmented with zinc tetroxychromate gave excellent adhesion to metal and freedom from blistering on soaking in salt or fresh water. A thin (0.5 to 1.0 mil) wash coat of this product alone did not give protection for an extended period of time, but did improve the performance of the paint system subsequently applied. The outstanding property of the wash coat was its adherence to metal and its improvement of the adherence of the primer. Excellent adhesion was obtained on every metal to which it was applied, which included mild steel, stainless steel, unanodized or anodized aluminum, zinc or galvanized iron, copper, tin, and cadmium plate. Primers that had no adhesion to metals were used over the wash coat and the resulting system had excellent adhesion.

Blistering and peeling of paint films on ship bottoms have been rated the principal causes of failure in service, but there was very little information on the reason for this occurrence. In order to understand this more fully, several laboratory tests were set up to study the underlying causes of paint failures.

16.3.3 Mechanism of Blistering

When steel is immersed in an electrolyte and made the cathode in an electrolytic circuit, hydrogen is liberated at the metal surface, while the metal itself is protected from corrosion. This is demonstrated simply by immersing a bare steel panel connected with a strip of zinc in sea water for a few hours, when numerous small bubbles of hydrogen will be observed on the steel surface, and the steel will remain bright and uncorroded. If a permeable membrane, such as paint film, be introduced between the steel cathode and zinc anode, the loss of hydrogen ions to form molecular hydrogen will tend to produce a corresponding increase in hydroxyl-ion concentration at or near the steel surface, thus increasing the pH under the paint film and setting up conditions favorable to osmosis. The paint film may thus be subjected to either hydrogen gas pressure or liquid pressure, or both, and blisters will form unless the adhesion of the film to the steel surface is sufficient to balance the opposing forces.

This condition was caused in the laboratory by coating sandblasted steel panels on both sides, connecting them to strips of zinc and immersing them in sea water. Control panels similarly coated but not connected with zinc were also immersed and a third set was connected with copper strips. In every case where blistering developed it was much more rapid and severe on the zinc-coupled panels than on either of the other sets, though the steel beneath the blisters was found to be bright and clean. The copper-coupled panels, as expected, showed very bad corrosion at isolated spots where there were any imperfections in the film and all the blisters were filled with rust. The Navy 84 primer showed numerous small blisters in 24 hours on the zinc-coupled panels, but required 10 days to blister visibly on uncoupled panels. Vinyl chloride-acetate copolymer resins, pigmented with zinc yellow, which yield films of high wet strength and rather poor adhesion, showed no blistering coupled or uncoupled in two weeks, but were found to have loosened from the steel on the zinc-coupled panels and to have permitted corrosion to spread beneath the film on the copper-coupled and the uncoupled panels. Polyvinyl butyral resins, pigmented with zinc tetroxychromate, which yield films of good strength and adhesion but only fair resistance to moisture, showed no blistering or corrosion on any of the panels, except a few spots on the edge of the copper-coupled panels. These latter primers remained in excellent condition for three months, when the tests were discontinued, all other primers having failed badly in one form or another at the end of six weeks.

An especially interesting observation on several of the uncoupled panels was the formation of two distinctly different types of blisters as little as ⅛ in. apart. One type had bright metal underneath and contained a liquid alkaline to litmus. The other type was badly rusted underneath and contained a liquid acid to litmus. This illustrates the effects produced by local cells on surfaces, where the overall electrical potentials may be balanced, but still great activity exists at isolated small points.

These effects have practical significance since it is known that potential differences varying from a few millivolts up to 2 to 3 v do exist on steel bottoms. Thus a large portion of the area may be cathodic, where blistering is apt to be accelerated, while the remainder of the hull is anodic, where corrosion is most severe. For this reason it appears that panel testing should be revised to include this factor, which can be done by connecting zinc strips to one set of panels and copper strips to another. By such means the ability of a coating to withstand both cathodic and anodic conditions can be determined.

In order to determine the permeability of various coatings to ions, a paper extraction thimble was coated on the outside with the material to be tested. The thimble was then placed in a test tube where it was surrounded by an agar gel containing a suitable indicator, and a solution containing the ion under consideration was placed inside the thimble. Penetration of the ion through the coating was indicated by a change in color in the agar gel adjacent to the coating.

In one set of tests 1N hydrochloric acid was used in the thimble with methyl red indicator in the gel; in another set sea water was used in the thimble and 5-p-acetamino-phenylazo-8-hydroxyquinoline in a slightly alkaline solution was used as an indicator for magnesium. In both instances polyvinyl butyral showed remarkable impermeability, with polystyrene nearly as good. Phenolic films were not so good, and glyptal coatings failed very quickly.

16.3.4 Electrical Resistance of Coating as a Measure of Deterioration

Since it may be assumed that changes in the electrochemical equilibrium at the metal-paint interface are closely connected with the initiation of corrosion and blistering, and since such changes may be in progress long before their ultimate effects become visible at the outer surface of the paint film, it would be an advantage to be able to detect them immediately as they occur, thus making it easier to connect cause and effect.

It has long been known that corrosion or solution of a metal is accompanied by a change in its surface potential, which can be measured accurately in relation to a standard, such as a calomel electrode. This appeared to be a reliable and useful method of measuring the corrosion resistance of coatings in sea water, particularly since it had the advantage over most laboratory tests of approximating actual service conditions rather than being an "accelerated test" wherein abnormal destructive conditions prevail.

A specially designed electrometer was constructed which in principle was similar to the commonly used potentiometer systems. By the use of an electrometer tube as a null indicator in place of the usual galvanometer, coating resistance as high as 1,000 megohms could be determined with an accuracy of ±2 per cent. Since only a very small current flowed during the measurement, the coating was not subjected to any accelerated breakdown and the effects of polarization were negligible. For the purposes of avoiding contamination of the sea water and of eliminating junction potentials, a specially constructed sea water-calomel reference half-cell was used. This half-cell was identical with the commonly used calomel references except that sea water instead of a potassium chloride solution was used as the aqueous medium.

The cells as set up may be represented as mild steel-organic coating-sea water-calomel-mercury. The internal resistances of such cells may be determined by use of the electrometer. By subtracting the internal resistance obtained in a similar cell where the mild steel electrode is uncoated, the resistance due to the coating may be calculated. In the systems investigated, the internal resistance when the steel was bare was approximately 1,400 ohms, varying negligibly with the extent of the corrosion of the steel. Since the internal resistance of cells involving coated steel varied between 1 megohm and 1,000 megohms when protection was good, this 1,400-ohm resistance had to be considered only when the coating approached failure.

A thorough testing of coatings was not made with this equipment, but enough work was completed to demonstrate the reliability of this method of studying and evaluating organic coatings for protecting the hulls of seagoing vessels. Most of the results were obtained with clear coatings which cannot be used for predicting the relative behavior of pigmented coatings containing the same vehicles.

It was found that when the logarithm of the resistance in ohms of 1 sq cm of coating surface was greater than 8, it was accompanied by complete protection as long as the high resistance continued. A low or decreasing resistance (log R less than 6) was indicative of the onset of rust formation and film failure. With resistances between these two, the results were doubtful and depended on the trend of the change in the resistance. This method should be of considerable importance in furnishing information leading to the specification of better organic protective coatings for the hulls of seagoing vessels. By the proper control of variables, coating resistance studies should yield the quantitative effect on protectivity of such important factors as metal surface pretreatment, drying time and aging, pigmentation, inhibitive constituents, solvent, and thinner, as well as differences in the sea water encountered in service.

This method of study is apparently not limited to steel substrates and a sea water environment. In general, coating resistance studies may be used with any metal substrate on exposure to all types of natural waters and special aqueous solutions. A limited number of tests with coatings on magnesium substrates exposed to sea water indicated that such a test may be valid with magnesium and magnesium alloys. This is important, since such substrates have been found to corrode seriously in practice with little or no visible protective coating rupture. The results obtained to date show that although the protective coating appears to be in good condition its resistance is considerably lowered when the destruction of the magnesium is proceeding.

From a more academic standpoint, resistance measurements may have some value in studying the rate of growth and distribution of corrosion products on certain uncoated metal surfaces immersed in aqueous media. Preliminary results obtained in the study of uncoated magnesium partially immersed in sea water suggest that the resistance at the metal-liquid interface increases (0 to approximately 1,000 ohms per sq cm) with the formation of the corrosion product. Further work is needed to substantiate this possibility.

In addition to studies on coated metal surfaces continuously submerged in aqueous media, resistance measurements may be used in conjunction with intermittent immersion and with atmospheric protective coating tests. In the latter case conditions should allow for the occasional submersion in an aqueous medium required for taking measurements.

16.4 ANTIFOULING COATINGS

16.4.1 Introduction

It is essential during war conditions to keep the bottoms of naval vessels free of fouling in order to avoid the consequent serious loss of speed, maneuverability, and fuel consumption. For example, the saving in fuel for ship propulsion has been variously estimated at 20 to 50 per cent in the case of a clean bottom as compared to a fouled bottom. Also the bottom coatings should have a long life and be easy to apply so as to reduce the frequency and time of docking to a minimum.

At the beginning of World War II, the Navy coating practice consisted of brush or spray application of an anticorrosive primer, which required several hours to dry, followed by two coats of a varnish type of antifouling paint, containing rosin, coal tar, and toxic ingredients such as mercuric oxide and cuprous oxide. Application of these coatings was easy, but considerable time was lost in waiting for the various coats to dry. Partly on account of this, and partly because a thicker antifouling film than could be obtained by this cold application of solvent-containing paints was desirable, the antifouling coating was changed to a "hot plastic" containing no volatile solvents and applied by means of electrically heated spray equipment at a temperature of 250 to 300 F. The hot plastic consisted of a mixture of paraffin, phenol-formaldehyde modified resin, mercuric oxide, cuprous oxide, and Paris green. This coating, designated as Mare Island plastic No. F-142, had excellent antifouling properties and had the further advantages of hardening very quickly on cooling, so that a ship could be returned to the water as soon as the coating job was completed, and of easily building up a heavy coating to a thickness of 50 to 60 mils.

This coating had disadvantages, however, which served as a basis from which to start in the development of improved coatings. The necessary heating to 250 to 300 F involved rather troublesome equipment; it was difficult in cold weather to obtain a smooth homogeneous film because of the rapid chilling of the hot compound when it touched the cold steel; rapid chilling caused excessive shrinkage and poor adhesion; in hot summer weather, the material was apt to remain too soft and produce excessive sagging. Thus temperatures above 100 F or below 50 F were outside the range of best conditions for application.

The testing of hundreds of toxic materials for antifouling paints over the past century or longer has indicated that copper and mercury compounds are the most effective. These materials are sufficiently effective to prevent fouling over periods of two years, and failure is generally due to the vehicle rather than to the toxic.

The vehicle must be of the proper composition to permit the toxic to be at the surface in the correct amount. The use of a highly impermeable vehicle may seal the surface so well that the poisons are prevented from diffusing to the surface, or the use of too permeable a vehicle may permit leaching of the poisons so rapidly that the supply is quickly exhausted. Thus, quantitative measurements of the rate of leaching should provide a reliable means of determining the probable behavior of a paint and of studying the effects of variations in vehicle composition, pigmentation, poisons, and other important factors.

16.4.2 Copper Leaching Tests

At the beginning of this work it was tentatively assumed that the toxic copper or mercury compounds must be soluble in sea water and must be leached out of the supporting matrix gradually enough and at just the right rate to maintain toxicity on the surface of the film or in the thin layer of slime that collects on all objects immersed in the sea. This was the theory generally advanced in most of the previous publications on the subject of fouling prevention. However, it was difficult to imagine a toxic layer consisting of an actual solution of copper salts that would not be washed away too rapidly, even by the slow movement of water in a quiet harbor, to permit an effective concentration of copper to be maintained continuously at or near the surface. Also, it seemed probable that if leaching should occur at a rate rapid enough to produce any appreciable concentration of soluble copper, the supply would soon be exhausted. If fouling prevention must depend upon soluble copper being continuously released from the paint film to the surrounding ocean, this apparently means that a paint composition must be devised which will be capable of releasing soluble copper at a rate neither too slow nor too fast, and that the rate must not vary greatly over long periods of time under service conditions where changes of temperature, speed of the ship through the water, and chemical composition of the water in different harbors, as well as progressive changes within the structure of the paint film, operate against the possibility of securing and maintaining any standardized or controlled rate of leaching. This seems an almost impossible assignment unless there are other factors not explained by the soluble poison theory.

It was suggested that certain forms of copper or mercury compounds which are almost insoluble in sea water of *p*H 7.5 to 8.5, and which are now known to be formed at the paint surface by the chemical action of aerated sea water upon the more soluble metal compounds in the paint, may be the effective toxic agents. Evidence to support this theory was found in the fact that certain paints containing red cuprous oxide, finely divided metallic copper, cuprous chloride, or other soluble forms of copper, when immersed in aerated sea water, gradually formed a tightly adhering deposit of green or gray copper salts which were almost completely insoluble in sea water at a *p*H of about 8.0.

This theory obviated the necessity for a continuous leaching and washing away of soluble copper from within the film at a high rate in order to maintain a toxic concentration at or near the paint surface, but made necessary a chemical and physical structure in the paint film favoring the accumulation on its surface of insoluble copper salts in the form of a tightly adhering deposit or colloidal precipitate. Careful observation of those antifouling paints which yielded the most effective results on surface ships, including the Navy F-142 hot plastic and the Navy 15 R.C., disclosed that a green or gray deposit did form on the surface and that the time required for its formation varied greatly with the composition of the vehicles and the temperature. On a number of experimental paints exposed on panels at Miami, Guantanamo, and San Juan, good fouling protection was invariably accompanied by a visible accumulation of green or gray-green copper salts on the paint surface. All other paints which retained their original red or brown color fouled badly. The exact composition of the copper salts was not determined, but it was believed they were a complex mixture of basic cupric salts, possibly containing some copper oxychloride and basic copper carbonate.

To determine quantitatively the amount of copper leached out of a given paint composition and to study the factors controlling the formation of the green deposit, a simple laboratory technique was devised. The paint under examination was applied to the outside surface of a glass test tube, ¾ x 6 in., by dipping to a depth of 2 in. and was allowed to dry at room temperature in an inverted position. The average area coated was 30 sq cm. The coated tube was then suspended and centered by supports in a larger test tube, 1½ x 6 in., containing 50 cu cm of aerated sea water having a *p*H of 8.0. To accelerate the action, the temperature was maintained at 40 C by storing the samples in an oven with air circulation. At intervals of seven days, the water was removed along with any loosely adhering precipitate. Fresh sea water was then added and the cycle repeated as long as any significant changes occurred. The water was analyzed for both soluble and insoluble copper by a colorimetric method employing sodium diethyldithiocarbamate in a 0.1 per cent aqueous solution as indicator.

As the extraction cycle was repeated, it was noted that in the case of some paints, the original color of the paint gradually became masked by a chalky deposit which was firmly adherent. After varying periods of time, this deposit became thicker until it was visibly green or gray in color. With most types of finishes, the accumulation of a visible deposit coincided with a marked reduction in the quantity of copper released, leading to the belief that the insoluble deposit sealed the pores of the paint surface and so prevented further substantial extraction of copper. Removal of the green salts by sanding or scraping usually caused a large increase in copper released during the next cycle. The green coating could also be quickly removed by immersion in dilute acid, and subsequent extraction cycles yielded results closely resembling the original values.

A striking difference was noted, not only in the quantity of copper released, but also in the physical characteristics of the green precipitate from various paint compositions. In some cases a very fine-grained precipitate was formed which adhered tenaciously to the paint surface and could not be removed without drastic abrasion. In other cases, the precipitate did not adhere to the paint surface, but settled to the bottom of the tube, or could be removed easily by a stream of water or light rubbing. In some cases neither a green film nor an appreciable quantity of extracted copper was obtained. Some paints formed only a microscopic or iridescent surface layer while a few became green all the way through the paint. These wide differences were obviously connected directly with the composition of the paint vehicle, for the results with any given paint could be duplicated consistently or caused to change greatly by addition of other ingredients.

Study of leaching rate curves for various types of copper-containing paints showed that a considerable period of time elapsed before a paint reached a condition of approximate equilibrium with its environment and came to a fairly constant leaching rate which could be maintained for a number of months. The time required to reach this equilibrium and then the further time that the constant leaching rate could be maintained were no doubt closely related to both the physical and chemical structure of the paint, but no reliable means were found to calculate or predict just how long any given paint composition should remain completely resistant to fouling. Comparison of leaching rates and results of actual exposure tests indicated that sustained leaching rates of 8.2 mg of metal per 1,000 sq cm of surface per 24 hours or above were usually accompanied by complete absence of fouling; the range between 4.4 and 8.2 was somewhat doubtful; and leaching rates below 4.0 were accompanied by definite fouling. However, the correlation between numerical leaching rates and actual fouling resistance was not conclusive. In several instances, very high leaching rates entirely failed to prevent fouling, while, on the other hand, several paints with very low leaching rates remained entirely free of fouling. Apparently the chemical composition at the paint surface was more significant than the numerical values for the quantitative amount of copper which escaped into the sea during any particular period. In other words, it appeared that barnacles may be less affected by copper actually in solution in the water surrounding a ship or test panel than by the almost insoluble precipitated copper salts which were formed, under favorable conditions, on the paint surface and in the pores of the paint near the surface to provide high surface concentrations of copper.

16.4.3 Antifouling Coating Formulations

Incorporation of rosin in antifouling compositions greatly increased the amount of copper extracted during the first few weeks of immersion, probably by increasing the permeability of the vehicle to sea water and assisting the diffusion of copper from the interior of the film. Also rosin tended to increase adherence of the insoluble salts to the surface, an effect that was considered to be very desirable. High proportions of rosin, however, tended to reduce the toughness of the film and made it more brittle, probably increasing its tendency to crack and erode. Therefore, while rosin was a valuable aid in making the copper within the paint film more readily available in an active toxic form, there was a limit to the amount that could be used without producing poor film properties and causing failure of the paint by cracking or erosion.

The problem of producing a paint composition capable of forming a toxic copper deposit on its surface must also take into account the ratio of copper to binder, and the amounts and types of inorganic fillers or pigments used. In general, a high ratio of copper tended to increase the amount of copper that could be extracted by sea water, at least during the first few weeks of immersion, because the protective layer of binder surrounding the copper particles was thinner. It might be expected that the highest practical ratio of copper would be most desirable, but the limiting factor was the toughness or bonding strength of the binder. Some of the more brittle, weaker types of binders, such as rosin, coal tar, or waxes, would not tolerate more than about two parts of total inorganic material to one part of binder without producing films that were too weak or friable to withstand the erosive action of the waves. On the other hand, the use as binders of the extremely tough, elastic types of products represented by the high molecular weight polymers of vinyl chloride, vinyl acetate, styrene, isobutylene, methyl or butyl methacrylates or other similar materials, permitted the incorporation of as much as four or five parts of inorganic material to one part of binder without producing an excessively weak or brittle paint film.

Certain types of nontoxic inorganic fillers or extenders apparently had considerable value in these high pigment-binder ratios for several reasons. They replaced a considerable proportion of the expensive toxic copper or mercury salts and conserved the supply of these strategic metals. A more important consideration was their effect upon permeability of the film to sea water. The use of a pore-producing filler such as diatomaceous silica had the desirable effect of increasing the rate of diffusion of copper salts to the surface and also of improving the adhesion of the insoluble copper salts formed there in contact with sea water. Thus it became possible to use a highly water-impermeable type of binder, which resisted for long periods of time the softening and disintegrating action of sea water, and still retained a satisfactory rate of diffusion of toxic materials for maintaining an antifouling surface.

Hundreds of panels were made with various anticorrosive and antifouling systems and exposed at Miami, Florida, and Mare Island, California, all of which appeared satisfactory in preliminary laboratory tests. Quite a few systems remained perfect at the end of a year and several for eighteen months. Probably these finishes would have given protection for a longer period of time, but they were removed for examination of the film, the metal under the film, and for exhibition.

The vehicles used in the exposure tests included a copolymer of vinyl acetate-chloride, polyvinyl butyral, polyvinyl acetate, chlorinated rubber, polystyrene, chinawood oil phenolic resin varnish, dispersion resin, rosin, and zinc resinate. The pigments used were copper oxide, copper chloride, copper resinate, mercuric oxide, and extenders.

The oleoresinous binders have given good results in primers and topcoats, but some advantages found in high-polymer binders made them appear more attractive. High polymers were tough and could be loaded highly with pigments without becoming brittle. Their drying time was not appreciably affected by temperature since they depended only on solvent evaporation and not on oxidation or polymerization for hardening. Their films underwent very little change on exposure to sea water, which was an important factor in that the condition of a film that was saturated with water was more important than its actual water transmission. A film that retained its tough, leather-like properties when saturated with water gave more permanent protection than one with high water impermeability but which became weak when saturated. Successive coats of polymers could be applied within a short time interval, even in freezing weather, as a small amount of residual solvent did not influence the ultimate integrity of the film. In fact, those polymers that were alcohol-soluble hardened about the same in water as they did in air.

Six panels remained in good condition after eighteen months of exposure. Formulas and exposure results of these six coatings are shown in Tables 1 and 2. These panels were for test of antifouling coatings only. A baked phenolic primer was used so as to avoid any primer failure.

In the tests made, the best results were obtained using a binder of polyvinyl butyral or copolymer of vinyl acetate and chloride. There was not much difference in the behavior of the two resins for total submersion, but the butyral resin exhibited less cracking at tide level where the film dried out periodically. This choice of binding agent was applied to both anticorrosive and antifouling paints, and by using the same type of binder for both coatings, better adhesion was obtained between coats. A complete coating system giving excellent results was:

TABLE 1. Exposure results of six tested antifouling coatings.

Panel No.	Composition*	Exposure Period and Performance Ratings						Relative overall rating	Comments 18 months' exposure
		12 months		18 months					
		F.R.**	C.R.†	F.R.	C.R.	A.F. Film‡	A.C. Film§		
2	4.2 Vinylite VYHH 12.5 Rosin 83.3 Cu_2O Sand granules in 2nd primer coat	100	100	100	100	98	100	2	Slightly rough owing to sand granules. Few small cracks in A. F. film. Remains sound after drying out. Adherent green surface deposit.
3	Same as panel 2 without sand granules	100	100	100	100	98	100	1	No defects except in edge trim. Remains sound after drying out. Adherent green surface deposit.
13	4.5 Vinylite VYHH 4.5 Rosin 45.5 Cu_2O 45.5 HgO	100	100	100	100	98	100	1	No defects except in edge trim. Cracks and peels after drying out. Adherent olive drab surface deposit.
14	8.3 Vinylite VYHH 8.3 Rosin 83.3 Cu_2O	100	100	98	95	95	95	3	1 spot peeled $\frac{3}{4}$ in. from edge. Cracks and peels badly after drying out. Adherent mottled green deposit.
23	57.3 Navy 15 R.C. 5.1 Vinylite VYHH 25.8 Cu_2O 11.8 Celite	97 Very few barnacles	100	90	100	90	95	4	Peeling of A.F. coat at one corner. Few cracks after drying out, but does not peel. Adherent fawn color surface deposit.
29	59.4 Navy 15 R.C. 5.2 Chlor. rubber 21.1 Cu_2O 13.0 Celtite	100	100	90	85	80	80	5	Peeling and corrosion at all corners. Cracks and peels badly after drying out. Adherent fawn color surface deposit.

* All these paints appeared physically sound on removal from test racks, but after drying out several days in the laboratory some cracked and peeled as noted above.

** F.R. = Fouling resistance.

† C.R. = Corrosion resistance.

‡ A.F. Film = Physical condition of antifouling coat.

§ A.C. Film = Physical condition of undercoats.

TABLE 2. Important formulations.

	Per cent by weight paint	Per cent by weight dry film
WP-1 *Wash Primer*		
Base A		
Vinylite XYHL resin (butyral)	8.96	38.2
Zinc tetroxychromate	8.54	36.4
Celite 165–S	1.42	6.1
Monarch No. 71 black	0.08	0.3
Butanol	81.00	....
	100.00	100.00
Thinner		
Phosphoric acid (85 per cent H_3PO_4)	15.2	19.0
Ethanol-denatured	84.8	.....
	100.0	100.0
Mix in proportion of 77.2% Base "A" and 22.8% Thinner within 24 hr of use.		
P-10 *Primer*		
Vinyl chloride-acetate resin VAGH	17.0	40
Red lead	25.5	60
Methyl ethyl ketone	16.9	....
Methyl isobutyl ketone	20.3	....
Xylol	20.3	....
	100.0	100.0
AF-14 *Antifouling Paint*		
Vinyl chloride-acetate resin VYHH	5.9	8.3
Rosin	5.9	8.3
Cuprous oxide	59.4	83.4
Solvesso No. 1	14.4	....
Methyl isobutyl ketone	14.4	....
	100.0	100.0

For wood-bottom ships good results were obtained by applying AF-14 directly to the wood. Another formulation (AF-22), containing metallic flake copper in place of cuprous oxide, was developed because of the short supply of the latter, and found considerable use on wood vessels of both Navy and Army.

16.5 PAINT REMOVAL AND METAL CLEANING

16.5.1 Chemical Paint Removers

Improved paint removers were requested by the Navy for stripping of paint from aircraft. It was desired that the material be nontoxic and noninflammable, and since it was to be used largely on aluminum, it was necessary that it be inert to this metal.

About 50 solvents were examined for stripping ability on the listed Navy paints. Lacquer L-12a: nitrocellulose base; Primer P-27B: zinc chromate, reduced congo, alkyd and phenolic resins; Enamel E-5: phthalic base; Enamel E-6: glyceryl phthalic base (baked); Varnish V-10c: phenol-formaldehyde resin, tung oil; and Varnish V-11f: glyceryl phthalate base. The commonly used acetone-benzene mixture (50-50) was used for comparison.

The most efficient paint strippers, without regard to other properties, were found to be (1) cyclohexylamine, (2) morpholine, (3) mesityl oxide, (4) methylene chloride, and (5) dichloroethylene. Of somewhat less effectiveness were ethylene dichloride, propylene dichloride, trichloroethylene, 1,1,2-trichloroethane, and cyclohexanone. Of these materials cyclohexylamine and morpholine are harmful to the skin and irritating when inhaled, while cyclohexylamine appeared slightly corrosive to aluminum. Mesityl oxide has too low a flash point (33 F), is irritating to the skin, and is probably toxic. Ethylene dichloride and propylene dichloride both have flash points of 21 F. Cyclohexanone is less active than most of the others as a paint stripper. The best material from all standpoints was methylene chloride.

Various combinations of materials were tried, some of which were fairly satisfactory. The best composition was methylene chloride thickened with a small amount of cellulose acetate and rubber, previously dissolved in a mixture of 10 per cent ethanol in methylene chloride.

Compositions very similar to this were obtainable commercially under the trade names Pyrox Remover (Pyrox Chemical Corporation, Long Island City, New York), Devoe-Raynolds Remover (Devoe and Raynolds Company, Inc., Philadelphia, Pa.), Saf-Te Remover (Wilson-Imperial Company, Newark, N. J.), I.C.D. No. 9 (The Billings-Chapin Co., Long Island City, N. Y., The Glidden Co., Reading, Pa., and Telton, Sibley and Co., Philadelphia, Pa.).

Other high-flash formulations with satisfactory paint stripping properties were:

1. Cyclohexanone (flash point 127 F)	80 per cent by volume
Methylene chloride	10
Carbon tetrachloride	5
Tricholoroethylene	5
Flash point of mixture 140 F	
2. Hexalin acetate (flash point 140 F)	80
Methylene chloride	20
Flash point of mixture 150-155 F	

16.5.2 Mechanical Cleaning Methods[14]

Early in the work on the development of improved coatings for ship bottoms it became apparent that a common cause of paint failure was improper cleaning of the metal surface before application of the paint. In October 1941, the three most important factors concerned with the improvement of coatings for the Navy were outlined in the order of importance as (1) cleaning and preparation of metal surface, (2) anticorrosive and protective coatings, and (3) antifouling compositions. A recommendation was made to the Navy that sandblasting or steel shot-blasting be used for cleaning ship bottoms. Since dry sandblasting was objectionable because of the danger of abrasive damage of ship mechanisms, it was suggested that wet sandblasting be developed for this use. Up to this time the hulls had been cleaned by handscraping or by air-operated chisels, followed by wire brushing. This method did not obtain a surface in proper physical condition to secure best results from the anticorrosive primers applied.

In 1942 an investigation of methods of cleaning ship hulls was undertaken. Five possible cleaning methods were considered: chemical, electrolytic, flame, abrasive, and impact. Chemical cleaning was not considered satisfactory because of the character and size of the dock and ships. Investigation showed that it would be an expensive and time-consuming job and that the method of application and removal of the chemical might prove to be most troublesome and injurious to dock structure and to the workmen.

The best electrolytic methods for cleaning metals required submersion in a tank of acidic electrolyte solution. This would be impossible with a ship, and portable apparatus would be difficult to design. Sea water could perhaps be used as the electrolyte while the ship was in the fitting-out basin, but its use would be limited to that part of the operation. There was also doubt as to the penetrating ability of the electrolyte on the practically impervious layer of paraffin base antifouling paint on the hulls of most naval vessels.

The flame method of cleaning hulls was not seriously considered because the Navy objects to the use of open flames in dry docks. It was doubtful whether flame would work well because of the low melting point and inflammability of the plastic paint used.

Abrasive methods such as revolving sand disks were fairly suitable for small boats and were in use by the Navy for this purpose. For large ships, however, the time allowed for recoating was so sharply restricted that this method was considered too time consuming.

Impact methods were believed to be the most promising field for investigation, and two systems were examined: (1) impact from a mechanically operated member, and (2) impact from moving solid particle, such as sand or steel.

Of the mechanically operated tools available, the best appeared to be a device manufactured by the Aurand Manufacturing and Equipment Company of Cincinnati, Ohio. This tool could be driven by either a pneumatic or electric motor, the former using a power source more available in Navy dry docks. The cleaning action was obtained by revolving a master wheel at high speed. About the periphery of the master wheel were groups of cutter wheels, shaped like milling cutters. The wheels were loosely fastened so that they flew outward under the action of centrifugal force. Thus the cutters, when permitted to make contact with a metal surface, flaked off such coatings of rust or paint as were present.

Laboratory tests with this tool on a variety of surfaces were promising enough so that practical tests were made on several cruiser hulls. One hull was badly pitted and corroded with a thick tough layer of black iron oxide, and over this surface was a heavy layer of Navy antifouling plastic. On this hull the Aurand tool was unsatisfactory because of the very slow rate of cleaning and because it failed to remove entirely the heavy oxide layer. These results were in contrast to those obtained on two other cruiser hulls which were in better condition. On the other cruiser hulls the Aurand tool produced a cleaner surface than was obtained with power chipping and at a somewhat faster rate.

Tests with the Aurand tool were made on the superstructure and interior of one of the cruisers, and the tool appeared to be very satisfactory in rate and quality of work for removing paint and cork insulation from light partitions and bulkheads. It was believed that this type of cleaning instrument deserved further study and use for this type of work.

For a study of wet sandblasting the Hydro-Blast system was investigated. This system, produced by the Hydro-Blast Corp. of Chicago, was used for cleaning foundry castings and in its commercial form was not portable. An examination of the unit showed, however, that it could be broken down into a relatively simple system that could be used in a dry dock for cleaning ship bottoms. The unit as modified for tests at the Brooklyn Navy Yard consisted of a high-pressure pump driven by a gasoline engine, which, together with a water reservoir, was mounted on a four-wheel chassis. A specially designed portable sand hopper was used as a source of abrasive. Potassium dichromate was introduced in the water stream to inhibit the formation of rust on the cleaned steel plates.

Tests showed that this equipment produced a very clean surface, but a number of mechanical changes would have been necessary to make it satisfactory.

Mare Island Navy Yard had been working on wet sandblast methods of cleaning ship bottoms and developed a vapor sandblast which proved entirely satisfactory in producing an exceptionally clean surface at a rapid rate considerably in excess of power chipping. For this reason the NDRC investigation was discontinued.

16.6 BACTERIAL EFFECTS ON METAL CORROSION AND PAINT DEGRADATION[21,24,25]

16.6.1 Introduction

As a result of a study[1] of pipe line corrosion, it has been reported that of the factors responsible for corrosion, such as stray current electrolysis, acid, carbon contact, and differential aeration, anaerobic corrosion is second in importance only to stray current electrolysis. Anaerobic corrosion is the result of sulfate-reducing bacteria and is not self-stifling such as certain types of aerobic corrosion where the products exert an insulating effect. Under conditions favorable for anaerobic corrosion in soil, failure of steel pipes 0.2 in. thick has been noted in seven to eight years.

It was determined that little information existed concerning the extent or severity of anaerobic corrosion of metals in the sea, although some scattered accounts suggested the possible destruction of metal by this type of corrosion. When these were considered with the information on corrosion in soil, it was concluded that the possibility of sulfate-reducing bacteria being an important factor in the corrosion of ship hulls warranted investigation.

Sulfate-reducing bacteria have the capacity of reducing sulfate to sulfide. During the development of these bacteria in soil, in sediments, and in water containing even small amounts of iron, the substrates turn black because of the formation of FeS. If there is an excess of sulfide over that combined with iron and with certain other cations, an odor of free hydrogen sulfide can be detected. One of the most characteristic features of locations where these bacteria develop is the presence of black iron sulfide, or the odor of hydrogen sulfide, or both. Reduction of sulfate is characteristic of this special group of bacteria; in fact, no organisms are known other than those belonging to this group which are capable of reducing sulfate to sulfide. These organisms are small curved rod-shaped bacteria, sometimes spiral-shaped. Their average size is 4×1 microns. They have been described under the generic names Spirillum, Microspira, Vibrio, Desulfovibrio, and Sporovibrio, all of which refer to the same group of organisms. A few species have been described, but these are all alike morphologically and differ only in the organic materials upon which they are able to grow. One of the most common species is the one named Sporovibrio Desulfuricans.

These bacteria are particularly active in sediments of sea water and brackish waters; they are also widely distributed in terrestrial deposits. They can be recovered from agricultural soils as well as water-logged soils, but are active only under strongly reducing conditions. They are commonly encountered wherever cellulose and plant materials undergo decomposition under anaerobic conditions. The sulfate-reducing bacteria do not develop on the cellulose directly, but on the products formed during the anaerobic decomposition of cellulose by other bacteria, including organic acids and alcohols. These bacteria are responsible for the large amounts of sulfide in the Black Sea (5 to 10 ppm H_2S at a depth of 350 to 1,150 fathoms) and for its black color, which is due to FeS. They are the principal source of the black iron commonly encountered in muds of tidal basins and marine sediments.

The prevailing theory concerning the corrosion process was based on the work of von Wolzogen Kühr, who established in 1934 the relationship of sulfate-reducing bacteria to this process. Experimental evidence was submitted to substantiate the theoretical considerations. The following reactions are involved:

$$\left.\begin{aligned} 8H_2O &\rightleftarrows 8H^{+} + 8(OH) \\ 4Fe + 8H^{+} &\rightleftarrows 4Fe^{++} + 8H \end{aligned}\right\}\text{Anodic solution of iron}$$

$$CaSO_4 + 8H \rightarrow H_2S + 2H_2O + Ca(OH)_2 \quad \text{Depolarization}$$

$$\left.\begin{aligned} Fe^{++} + H_2S &\rightleftarrows FeS + 2H^{+} \\ 3Fe^{++} + 6(OH)^{+} &\rightarrow 3Fe(OH)_2 \end{aligned}\right\}\text{Corrosion products}$$

$$4Fe + CaSO_4 + 4H_2O \rightarrow FeS + 3Fe(OH)_2 + Ca(OH)_2 \quad \text{Summary}$$

The bacteria are of principal importance in the corrosion process by reason of their ability to remove cathodic hydrogen. Whereas this is effected by dissolved oxygen under aerobic conditions, it is brought about under anaerobic conditions by the sulfate-reducing bacteria which simultaneously reduce sulfate. The sulfide formed by this reduction reacts with part of the ferrous iron to produce ferrous sulfide. Ferrous hydrate is also produced and in larger amounts than ferrous sulfide; thus the corrosion products contain both ferrous sulfide and ferrous hydrate. Determinations made by von Wolzogen Kühr on corrosion products showed a ratio of total iron to FeS, which was somewhat below the theoretical value of 4 to 1.

16.6.2 Experimental Work

In order to obtain an idea as to the distribution of sulfate-reducing bacteria, samples of sea water were taken from Woods Hole and Buzzards Bay, Massachusetts; Brooklyn, New York; Barnegat Bay and Cape May, New Jersey; and Kure Beach, North Carolina; bottom sediments were taken from South Bristol, Maine; Woods Hole, Massachusetts; Cape May, New Jersey; Kure Beach, North Carolina; Miami and Biscayne Bay, Florida. All samples of both water and sediment gave positive test for the presence of sulfate-reducing bacteria.

Painted and unpainted steel panels were submerged in sea water at Woods Hole, Brooklyn, Barnegat Bay, Stone Harbor (New Jersey), Kure Beach, and Miami Beach. At all sites the corrosion of the bare steel panels followed the same pattern; the amount of fouling varied with the location. Aerobic corrosion began immediately upon submersion with the formation of red ferric hydrate. In fouling areas, the fouling organisms attached themselves very soon. Fouling was not so severe on the bare panels as on those coated with an inert paint, because the corrosion products tended to exfoliate and thus cause the fouling organism to break away. At the end of two to three months the corrosion products tended to adhere more tightly, and at about this time sulfide was first detected as a black material beneath the surface layer of red corrosion products. After four months[a] the entire surface of the panels was covered with the black sulfide-containing corrosion products with an overlying layer of ferric hydrate and, at most sites, fouling organisms. The sulfide layer was sometimes soft and resembled graphite, but at other times occurred as a hard crust. This black material could be readily removed from the metal and revealed a bright silvery surface with no adherent protective material. Without exception, the black corrosion products contained relatively large numbers of the sulfate-reducing bacteria. Upon exposure to the air the ferrous sulfide was rapidly oxidized to sulfate. A few ship bottoms were examined, and where the paint had failed the same type of corrosion was observed, red aerobic corrosion products acting as a barrier for the underlying black anaerobic corrosion products. The rate of corrosion of most of the bare steel panels decreased during the exposure period where anaerobic corrosion was assumed to be dominant, but was still fairly rapid.

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[a] This time varied with the site. The longest period was about 7 months at Barnegat Bay.

Since in all these exposure tests sulfate-reducing bacteria were found in abundance on corroding steel and a considerable amount of sulfide was found in the corrosion products, it was concluded that the bacteria were at least partly responsible for the metal destruction after the initial stage during which ferric hydrate alone is formed. The results failed, however, to establish an absolute relationship between development of sulfate-reducing bacteria and corrosion of steel exposed in sea water, and a laboratory experiment was set up in the hope of obtaining more specific data. The results, however, were not satisfactory. For these tests, steel panels were degreased with solvent, sandblasted, and sterilized in sea water. They were then exposed in sterile, non-aerated sea water; sterile, aerated sea water; non-sterile, aerated sea water; aerated sea water treated with 0.05 per cent mercuric chloride; and aerated sea water treated with 0.025 per cent Santobrite (sodium pentachlorphenate).

The results of this experiment did not duplicate results obtained in normal sea water exposure. The rate of corrosion was low in most cases and the corrosion products differed from those which prevail in the ocean. At no time was sulfide detected in the corrosion products, and while sulfate-reducing bacteria were recovered in the non-sterile sample, they were in much smaller number than would be expected if they were actively concerned with corrosion. The results are probably abnormal and do not reflect actual conditions. Somewhat more satisfactory results might be obtained if the experiment were performed in a laboratory near the ocean where the panels could be exposed to a continuous stream of fresh sea water.

Half of the painted panels which were exposed at the various sites were coated with an antifouling paint containing cuprous oxide over the primer, and the other half with a similar paint, except that it contained titanium dioxide pigment instead of the copper toxic, over the same primer. The inert paint quickly became fouled, and the slime and silt on these panels contained large numbers of sulfate-reducing bacteria, between 100,000 and 100,000,000 per gram of material. This was the range of numbers characteristic of the black sulfide-containing corrosion products where the sulfate-reducing bacteria were presumed to be active in anaerobic corrosion. There can be no question but that the sulfate-reducing bacteria are widely distributed in the ocean by means of fouling organisms and suspended particles of organic matter. These organisms may in fact be the principal agency by which these bacteria are distributed. There was no indication, however, that these bacteria affected the paint film, and where the paint was intact there was no corrosion of the underlying metal. Apparently the best preventive for anaerobic corrosion, as for aerobic corrosion, is an intact paint film.

While some sulfate-reducing bacteria were recovered from the slimes on the antifouling coatings, the numbers were generally much smaller than occurred on the inert paint. It was also observed that if the slime from the antifouling paint was used to inoculate a medium for growth of the bacteria, growth did not occur when the highest amounts of inoculating material were used, but that growth did develop at higher dilutions. This indicated that the copper was toxic to the bacteria until sufficiently diluted.

Slimes were examined which had formed on a number of copper-containing ferrous alloys after exposure at Kure Beach, North Carolina. The results indicated that sulfate-reducing bacteria were present and apparently developed even in slimes which formed on copper-containing metals, even though some of these slimes contained considerable copper. In general, however, the slimes with the higher amounts of copper contained fewer of the bacteria, suggesting that the copper exerted a toxic effect on the bacteria and tended to limit their development.

Laboratory tests showed that ferric oxide, red lead, and lead chromate were comparatively nontoxic, whereas metallic copper, brass, soluble copper compounds, and zinc chromate were toxic to the bacteria.

In order to obtain information regarding the relative rates of corrosion in strongly reducing sediments and in sea water, 12-ft steel rods and steel strips were exposed at Barnegat Bay and at Miami Beach. The specimens were driven into the bottom for a distance of 25 to 40 in. and most of the remainder was continuously submerged in the water. When examined at the end of six months, the portion which had been buried was bright with no adhering corrosion products, whereas the portion in the water was covered with red, crusted corrosion products. Measurements, however, showed that the loss of metal of the buried portion was as great as that of any portion of the specimen except for the section at the mud line, which was the most severely attacked.

Further tests were made at Cape May, New Jersey, in which steel panels were completely buried in the mud and duplicate panels suspended in the water directly above. In these tests the loss of metal was considerably greater on the panel in the water than on the buried duplicate.

16.6.3 Bacterial Attack on Paint

The panel exposure tests which have been described indicated that anaerobic bacteria had no noticeable effect on the paint films used. Antifouling paint surfaces would only rarely be under anaerobic conditions, and since aerobic bacteria would be most apt to attack these materials, tests were made in the laboratory to obtain some indication of the seriousness of paint degradation by bacteria. For estimating the extent of bacterial development, determinations of bacterial numbers proved to have very little value, but significant differences were indicated by the rates of oxygen depletion of the sea water in which the specimens were exposed. Tests of Navy paints indicated that paints 14 R.C. and 143E were most rapidly attacked, 42A was nearly as susceptible, and 15 R.C. and F84 the least affected.

Of paint constituents tested, Vinylite VYHH (copolymer of vinyl chloride and vinyl acetate), Halowax, coal tar pitch, and chlorinated styrene appeared to resist bacterial attack completely. Chlorinated rubber was slowly attacked, and with paraffin, alkyd, linseed oil, and two samples of phenolic resin the oxygen content of the water was rapidly reduced.

16.7 FIRE RETARDANT PAINTS FOR NAVY SHIPS[15]

A number of fires occurring on Navy ships at the start of World War II were aggravated by the burning of paint. This was particularly true of the paint on the bulkheads, which accelerated the spread of the fire from one burning compartment to adjacent compartments. One of the reasons for the fire hazard was the large number of coats of paint which had been applied to some ships without cleaning off the old paint. This resulted in an extremely thick layer which contributed to any fire that started. Laboratory studies made both by the Philadelphia Navy Yard and the NDRC showed that the thickness of the paint coating was the major contributing factor. This condition was quickly eliminated by removing all the old paint on interior surfaces and refinishing with not more than three coats of new paint. A further immediate improvement was made by reducing the amount of organic binder to about 20 per cent of the dry paint film and by substituting 10 to 20 per cent antimony oxide for other white pigments to produce a glazed or sintered residue at high combustion temperatures.

The apparatus used for testing the behavior of paints at high temperatures consisted of a welded steel box 13 in. wide by 13 in. deep by 19 in. long, arranged so that the test could be run either with or without a cover on the box to exclude air currents. The paint systems to be tested were applied to steel panels and inserted in the box in a vertical position approximately 3 in. in front of the gas burner, the uncoated side being next to the burner. In most of the tests two 6 x 12-in. panels were placed side by side, each coated with a different paint for comparison, or a 12 x 12-in. panel could be used with two or more different paint systems applied in strips. Each paint was tested with applications of one, two, or three coats, both with and without an anticorrosive primer coat.

The gas burner was connected to a cylinder of gas such as butane, propane, or butylene, and compressed air and the mixture adjusted to give a hot blue flame. With this arrangement it was possible to heat the panel to red heat in about 20 sec. It was considered desirable to bring the panels to a high temperature very quickly to simulate more closely the effects of a shell or bomb burst.

Observations during the heating period included the time and degree of flashing, the ability of the paint film to support combustion, and the tendency of the paint to blister and peel from the surface. A slight flash over the surface of a panel was not considered very bad, and in fact would probably be preferable to the gradual accumulation of sufficient unburned gases in a closed compartment to form an explosive mixture. It was noted that, with all paints examined, the tendency to flash was increased considerably as the number of coats of paint increased. With many paints, a single coat of paint produced no flash, two coats a very slight flash, and three coats a definite flash. As long as drying oils, alkyd resins, or similar organic materials were used as binders for the paint, it was recommended that no more than three coats be allowed to accumulate.

Paints which actually burn or support combustion after the gas burner is turned off were considered to be entirely unsatisfactory for use as interior coatings on ships. A number of experiments indicated that the proportion of pigment to combustible binder was a principal factor in the ability to support combustion. It appeared that a pigment content of at least 80 per cent by weight of the dry film was desirable to prevent burning. A small amount of antimony oxide (10 to 20 per cent of the total pigment) was also found desirable, apparently due to the relatively low melting point of this pigment and its ability to flux and retard the free access of oxygen to the organic matter of the film.

Paint systems which blister or peel during the high-temperature treatment were considered undesirable, even when they did not flash or support combustion. Some paints, when heated very rapidly to 1000 C in 20 sec, popped away from the steel, throwing off glowing hot particles with considerable force. These hot particles, even though not actually aflame, could presumably ignite papers or other easily combustible material in a compartment some distance away from the hot bulkhead.

During the testing it was noted that flashing, burning, and peeling were all accentuated when any of the new highly pigmented fire retardant paints were applied over the standard Navy 84 primer, which has a relatively high proportion of oleoresinous vehicle and low proportion of inorganic material. This was rather disturbing because this primer was usually applied to all new steel for the Navy at the mill to prevent corrosion during shipment and construction, and was also used as the standard undercoat for repainting older ships.

A means of overcoming this defect was found in the addition of aluminum flake to the Navy 84 primer before applying. With this addition, a marked reduction was noted in the tendency of the paint to peel, flash, or burn. This modification could be used without difficulty for repaint work after removal of all old paint, as the aluminum in powder or paste form could be stirred into the standard Navy 84 primer very easily by painters on the job. Best results were obtained by mixing 1 pound of aluminum paste containing 80 per cent aluminum with 2 pounds of Navy 84 primer, though smaller amounts of aluminum gave noticeable improvement over the 84 primer alone. Other metals such as zinc powder were also tried in mixture with the 84 primer. Some improvement was noted, though results were in no case as good as those obtained with aluminum.

Several different types of paint formulations were tested in single- and multiple-coat applications, both with and without the standard Navy 84 primer and its aluminum modification. These included the following materials:

1. A linseed oil base paint corresponding to the Navy No. 29 inside white.
2. The new alkyd resin base paint supplied by the paint laboratory at Philadelphia Navy Yard and covered by the Bureau of Ships Ad Interim Specification 52P22 (Int), November 1, 1942.
3. A water-emulsion paint based on a carbic anhydride-linseed composition as binder, with a high proportion of pigments:

Carbic anhydride linseed resin	15.3 lb
Titanium dioxide	42.4
Antimony oxide	10.2
China clay	32.1
	100.0

This was thinned with water to brushing or spraying consistency.

4. A proprietary water-emulsion paint widely used for interior wall finishing, Sherwin-Williams Kem-Tone.
5. A paint based on the flameproof Vinylite VYHH resin, a copolymer of vinyl chloride and vinyl acetate.

Vinyl chloride-acetate copolymer resin VYHH	15.3 lb
Titanium dioxide	42.4
Antimony oxide	10.2
China clay	32.1
	100.0

This was thinned with ketones or ketone-hydrocarbon mixtures to spraying consistency. It was not suitable for brushing.

The linseed oil interior paints previously used by the Navy, having a relatively low pigment content, flashed and burned very badly. Even two coats of the No. 29 paint were considered somewhat hazardous, while three or more coats provided a definite fire hazard.

The new 52P22 (Int) specification paint was definitely better in all fire retardant properties than the paints previously used. It did flash when two or more coats were applied and heated quickly to 1,000 C, but did not support combustion and did not blister or peel badly, except when applied over the No. 84 primer.

The water-emulsion paint (3 above) appeared equal to the Navy 52P22 (Int) paint in fire retardant properties and had the added advantage that it offered no fire hazard during application or storage aboard ship, as it contained no volatile inflammable solvents. Its disadvantages were that it could not be applied satisfactorily over rusty surfaces without the surface becoming stained by rust unless an oleoresinous or similar priming coat was first applied, and its resistance to scrubbing with strong soaps was not so good as was desired. It was also somewhat more difficult to spread evenly by brush application, though spray application yielded a very uniform and attractive surface appearance. Since fire prevention and control aboard ship is such an important matter, it would seem that further development work on the water-emulsion type of paint to overcome the defects noted while retaining the greater freedom from fire hazard, would be desirable.

Examined proprietary water-emulsion paints were especially poor in fire retardant properties, flashing and burning as readily as any of the oil base paints. This is probably due to the fact that they did not contain the high pigmentation necessary for fire-retarding usage.

Paints based on Vinylite VYHH resin, containing a high ratio of polyvinyl chloride, were the most resistant to flashing and burning of all the paints tested, providing they were thoroughly dry and free from solvents. Their resistance to peeling was not so good as several of the other paints, and there is the possibility that the acid fumes released at high temperatures might interfere with fire fighting by the ship's crew. Because of their excellent resistance to moisture, alkalies, and acids, these resins meet other service requirements especially well and because of their toughness, they may be loaded with an unusually high proportion of pigments without becoming brittle, thus reducing the amount of resin and objectionable fumes to a minimum. It is believed that they merit further consideration in any future work.

Chapter 17

CORROSION RESISTANT LININGS AND COATINGS

17.1 SUMMARY

SOME OF THE most important though least spectacular service problems were those having to do with surface protection by the use of proper coatings. In addition to the problems of ship-bottom coatings (Chapter 16), several miscellaneous coating requirements were analyzed and coatings selected.

One such problem was the need for a coating as a lining for chemical bombs and for shells to provide protection against mustard, lewisite, and certain smoke mixtures. The coating selected for this purpose was an unmodified heat-convertible phenol formaldehyde resin applied in a two-coat system to a total of 0.5 mil, and a CWS specification was prepared on the basis of the test data obtained. For coating the interior of bombs a method was developed which provided a uniform coat where spraying was impossible; the bomb was filled with coating liquid and drained at a controlled rate through an orifice of proper size.

An investigation of a number of possible linings for fuel and lubricant containers, especially the 5-gallon "Blitz" can, led to the selection of a phenol formaldehyde resin and the preparation of several QMG specifications covering their purchase.

The coating and sealing of Navy projectile base fuzes and primers developed to be an especially important problem because of the number of duds caused by penetration of water and ammonia vapors into the primer. After laboratory and surveillance tests covering adhesion, moisture penetration, air leakage, and corrosion, three coatings were selected and recommended for the fuze body. A chlorinated synthetic rubber plasticized with tricresyl phosphate was found to be markedly superior to the shellac formerly used as a primer sealant.

17.2 LININGS FOR CHEMICAL MUNITIONS

Pure mustard gas (*bis*-β-chloroethyl sulfide) is fairly stable in contact with ferrous metals, and when made from thiodiglycol offers no particular problem when stored in bare steel. Mustard made by the Levinstein process (H), however, is a mixture including some rather reactive components, and bursting of containers from internal pressure has occurred after short storage in warm climates. (See Chapter 10.) Decomposition of H is also accelerated by contact with steel, resulting in lowering of the vesicant content and deposition of a sludge which interferes with the ballistics of the shell or bomb. If methyl methacrylate thickener is used in the vesicant, the polymer is precipitated by the iron, causing wide variations in the viscosity of the filling. A coating was therefore desired which would protect the filling from deterioration resulting from contact with the container. From the standpoint of standardization and simplicity, it was desirable to use the same munitions for different chemical fillings. For this reason it was desirable that any coating used for H be satisfactory also for L (lewisite), WP (white phosphorus), FS (chlorosulfonic acid-sulfur trioxide smoke mixture), and FM (titanium tetrachloride).

Twenty-eight types of potentially useful materials designed for coatings, seaming compounds, and crevice fillings were examined. The coating materials were applied to steel panels and tested for flexibility and adhesion at room temperature and at −40 C. For the resistance tests the coatings were applied by dipping or spraying on steel rods. The rods were placed in glass bottles and sufficient of the agent was placed in the bottle to cover half the specimen. The bottles were placed in an oil bath at 65 C and periodic examinations of the coating were made. Viscosity determinations were made on the agents after filtering. Suitability of the coating was judged largely on the basis of a 30-day test. Variations in the method of application, film thickness, drying and baking conditions, and pigmentation were investigated and their effect on both impact resistance and resistance to the agent was determined.

The most satisfactory materials were the unmodified heat-convertible phenol formaldehyde resins. The stability of H in contact with steel coated with this material was as good as that in contact with glass. Sandblasting of the metal is preferable for adhesion but from the resistance standpoint is not essential. The coating should be applied as a two-coat system totaling approximately 0.5 mil. Adequate baking is essential, particularly on those resins which cure slowly, and the importance of making the correct allowance for thermal lag in the heavier masses of metal cannot be overemphasized.

One air-drying coating (SDO, an acetylene polymer of du Pont manufacture) was found which had adequate resistance. In those places where baking facilities are not available or where baking is impractical for some other reason, a two-coat system of SDO can be used. It requires air drying for the equivalent of four days under well-ventilated, normal temperature conditions or forced drying at about 140 F to an equivalent hardness. This finish should be applied over sandblasted steel at a total thickness of 0.5 mil. The second coat should be put on before the first is thoroughly dried to anchor it adequately.

Pigmentation of both the SDO and phenolic coating materials is desirable but not essential. Its value is largely in controlling consistency and flow, in facilitating inspection, and, in some cases, in reducing cost. Titanium dioxide should be made the basis of this pigmentation and a dark tinting color added to the first coat to distinguish it from the topcoat and to show up incompletely covered areas.

Favorable results were obtained in some laboratory tests with coatings and treatments based upon sodium silicate with or without the addition of corrosion-inhibiting salts. The results were too inconsistent, however, to warrant a recommendation of their use. Their cheapness and availability make them extremely attractive.

The two recommended coatings were resistant to H, L, and WP, fairly resistant to FM, but were not resistant to FS. Both FM and FS can be handled satisfactorily in steel if kept anhydrous.

Among the water-soluble materials tested, polyvinyl alcohol was found to have good resistance to H and L. It would not be expected to perform well as a coating for use with WP filling because of the water present. This material should be of particular interest as a seaming compound, and if seam closures are found necessary on any vesicant-carrying munitions, polyvinyl alcohol plasticized with glycerol seems to offer the most promise.

The following classes of coatings did not have adequate resistance to the vesicants:

Oil-modified alkyds.

Plasticized nitrocellulose.

Plasticized urea-formaldehyde resins.

Plasticized melamine-formaldehyde resins.

Plasticized phenol-formaldehyde such as Bakelite XR-15376.

Phenol-formaldehyde varnishes.

Acid-condensed phenol-formaldehyde resin.

P.F. resins from certain substituted phenols, such as Bakelite BL-8966.

Conventional varnishes from soluble gums.

Vinyl resins, with the exception of polyvinyl alcohol. Included were polyvinyl acetates.

Phenol-urea-formaldehyde combination, such as Plaskon's 389-4.

Acryloids.

Methacrylates.

All hydrocarbon polymers except SDO. These include Buna S, perbunan, and butadiene drying oil.

Combinations of alkyds with many of the above. All combinations tested were unsatisfactory.

Sulfur formaldehyde resin.

Thiokol.

Lactic acid resin.

Plasticized polyvinyl butyral.

Since most of the bombs for which linings were desired were constructed in such a way as to make spray application impossible, a new method of coating with a controlled film thickness was developed. This was a modification of the common industrial practice of dipping and withdrawing from the tank at a rate corresponding to the normal flow of the coating down a free surface. This eliminates runs and sags and provides for uniform thickness from top to bottom. It was found that the bombs could be satisfactorily coated by filling with the liquid and withdrawing it at a controlled rate through an orifice. By proper adjustment of per cent solids and orifice diameter, a uniform coating of any desired thickness can be applied. The rates of withdrawal and solids content to give a desired film thickness were established for a number of resistant coatings. Conditions for application to M47 and M67 bombs were worked out for a representative phenolic coating and SDO. Resistance to corrosion is not affected by the method of application (spray, dip, or flow coat) as long as films of proper and uniform thickness can be secured.

The recommendations resulting from this investigation were incorporated in specification CWS-196-131-207, which is a performance specification for phenolic coatings.

17.3 LININGS FOR FUEL AND LUBRICANT CONTAINERS

At the start of World War II no lining was applied to containers for gasoline and other petroleum products. This resulted in serious corrosion of the containers, and contamination of the liquid with rust, which plugged carburetors and fuel lines, seriously shortening the life of the container. This was especially true of the 5-gallon cans which were alternately filled and emptied, allowing contact of moist air with the surface between fillings. When linings were first used, a variety of types was applied. Some difficulty was encountered from peeling of the coating, which caused worse contamination than the rust, and some instances of increase in gum content of the stored gasoline. There was no record of which types of linings were proving unsatisfactory, and an investigation was therefore undertaken to determine what material should be specified for this use.

The coating properties required were good adhesion, flexibility, resistance to gasoline and moisture, and the ability to protect the coated metal from corrosion. The materials proving best on an overall basis were unmodified heat-convertible phenol formaldehyde resins even though they were not so flexible as might be desired. Although they do not have the general resistance characteristics of the phenolic coatings, several other products having better flexibility are equally resistant to gasoline. These include some products based on urea-formaldehyde and certain high molecular weight vinyl-type resins. Alkyds as a class are open to question, although certain of them might be satisfactory.

The surface preparation of the metal was found to be as important as the type of coating used. Sandblasting, after removal of oil and grease, was found to be the most effective means of surface preparation, giving considerable improvement in adhesion as shown by the definitely reduced tendency of the normally brittle phenolic film to flake off the surface.

As the thickness of the coating was increased, the flexibility decreased. With sandblasted metal a thickness of 0.70 mil can be used with safety on small containers (up to 5 gallons). Substantially heavier coatings could be applied to 16-gauge and heavier metal used in the fabrication of large containers. If the metal is not sandblasted, thinner coatings should be used.

A review of the conventional surface treatments indicated that they held no promise, and it appeared obvious that any effective solution would be found in only a coating, metal or organic, which would completely cover and protect the steel surface. Zinc-coated containers were considered unsatisfactory due to deleterious effects which would result on exposure to water, especially sea water. While other metals were considered, this method of protection was not investigated fully, since organic linings were thought to be preferable from the standpoint of application and availability.

Flexibility of the coating was tested by an impact tester which produced, by sudden impact, a dent in the test panel with the convex button on the coated side about 1.5 cm in diameter and 4 mm in depth. Flexibility was measured by the degree, or the absence, of cracking at the apex of the extruded button. Adhesion was determined by the ease or difficulty with which the film could be removed from the button by superficial rubbing.

Resistance to corrosion was determined by exposure of coated test panels which had been subjected to the impact test to a mist of 20 per cent sodium chloride solution. The measure of resistance was the number of days in the salt solution mist before failure of the film. Most products failed by loss of adhesion due to seepage of liquid under the film or by progressive corrosion from a weak point in the film. This test often showed up failure at the apex of the impact test button which was not obvious before the test.

The gasoline resistance and gum increase test consisted of immersion of small test panels coated on both sides in high octane aviation gasoline maintained alternately at 140 to 150 F for 7 hours and at room temperature for 17 hours for a total of 5 successive cycles, followed by a determination of the nonvolatile content.

Typical clear organic coatings tested included straight heat-reactive phenol formaldehyde resin, oil- and gum-modified phenolics, typical vinyl resins, heat-bodied drying oils, drying oil modified alkyds, alkyd-modified urea-formaldehyde resins, alkyd-modified melamine resins, nylon, several pyroxylin materials, shellac, polythene, a high molecular weight paraffin, methacrylate resin, and one or two other synthetic resins. Among coatings found to be acceptable from a gum increase standpoint were heat-convertible 100 per cent phenol formaldehyde resins, a 43 per cent dehydrated castor oil alkyd-modified urea-formaldehyde resin, a 43 per cent dehydrated castor oil alkyd-modified melamine resin, an experimental synthetic du Pont resin, and a high molecular weight vinyl resin. All others were either borderline cases or definitely poor. Of these coatings only the phenol formaldehyde resins were found to be outstanding in salt spray resistance, these coatings withstanding exposures of more than 45 days while the best of the remaining products did not last more than 10 days. The phenolics, however, were very brittle, causing fine cracks to result in the impact test, and corrosion of the steel substrate was found to occur at the apex of the button after 2 or 3 days in the salt spray test. Despite this weakness, however, the phenolics were very resistant to underfilm corrosion, and failures were confined to the immediate vicinity of the break in the film.

Pigmentation, although adding nothing to the essential resistance properties, was considered desirable because it was found to improve adhesion slightly and to facilitate inspection of coated containers.

An evaluation was made of a coating (Cox, U.S. Patent 2,200,469) formed by the electrolytic deposition on steel of mixed salts from sea water. This coating was not found to be satisfactory. The adhesion was poor, as shown by the impact test, and there was a tendency for the coating to powder off in gasoline. Several modifications of the coating method were tried in an attempt to improve the effectiveness of the coating, but none were as good as the patented process.

On the basis of this investigation, the Quartermaster Corps issued specifications OQMG-165, OQMG-167, OQMG-168, and OQMG-183 covering the phenolic coating and its application to fuel and lubricant containers.

17.4 SEALING OF NAVY PRIMERS AND FUZES

17.4.1 Introduction

The investigation described was undertaken because of the failure of a proportion of certain types of armor-piercing shells to explode. Desensitization of the time fuze was found to occur after the fuzes had been assembled in the shells, caused by the action of water and ammonia vapors from the ammonium picrate explosive. Penetration of moisture to the mercury fulminate in the primer mixture took place along with severe corrosion of the metal parts in the explosive train.

17.4.2 Fuzes

The principal laboratory tests employed in selecting the best coatings were tests of adhesion and of moisture permeability. Cadmium-plated steel, Duralumin, copper, and tin plate surfaces were coated, force-dried for 1 hour at 150 F, and aged for one week before being tested for adhesion with a knife. As a second test of adhesion, two pieces of aluminum foil were coated, cemented together with a thermoplastic adhesive at 120 C for 3 minutes, and then stripped apart in a Scott tensile tester. The force per inch width required to strip the foil was taken as the index of adhesion.

For determination of permeability, the coatings were applied 1.0-mil dry thickness on nonmoisture proof cellophane. These sheets were tested for permeability at 140 F with an atmosphere saturated with vapors of a 1.0 per cent ammonia solution on one side and phosphorus pentoxide as a desiccant and ammonia absorbent on the other side. Data obtained on uncoated cellophane provided a basis for calculating the permeability of the test coating.

A wide variety of coatings was tested and compared with the Navy specification OS-1433 (50-50 paraphenyl phenol-formaldehyde resin-chinawood oil as 52 per cent solution in xylol). The choice of plasticizers was limited to two — tricresyl phosphate (to improve flexibility and adhesion) and Aroclor 1254 (for minimum moisture permeability). The clear solutions were thinned to about 1.5 poises, which is a suitable viscosity for dipping or flowing. The consistency was further reduced for spraying. Titanium oxide, zinc oxide, and zinc yellow were tested as pigments (lead compounds cannot be used in the presence of ammonium picrate), with flake talc as extender. Zinc yellow was chosen as representative of corrosion inhibitors. The purpose of the pigmentation was to reduce permeability, inhibit corrosion, improve adhesion, and reduce the tendency of the coating to penetrate into the detent springs of the fuze. In general the pigments did not greatly help corrosion or adhesion, and the zinc yellow tended to increase permeability. However, a pigmented form of OS-1433 was included in the recommendations because of the reduced tendency to penetrate into the detent springs.

Coating tests on Mark 28 fuzes were carried out with a series of ten of the most promising unpigmented coatings, and with a second series of pigmented coatings. For the laboratory tests the fuzes were coated most successfully by dipping, and attempts to spray coat were generally unsuccessful. Film thickness was measured by a General Electric magnetic induction gauge. An air pressure of 40-cm water was applied to the inside of the sealed fuze to test for leaks. After coating by dipping for 2 minutes the penetration into the detent springs was noted by testing the action of the springs when the nosepieces were revolved up to 3,000 rpm. Of the ten unpigmented coatings, eight penetrated into the detent springs; all the vehicles could be made satisfactory in this test by pigmentation.

Several hundred fuzes were tested in a humidity cabinet at 140 F in contact with vapors of 1 per cent ammonia solution in water. Pellets of weighed desiccant were placed in the sealed fuzes and removed for weighings at intervals of two weeks. Although the consistency of the data obtained in these tests is not good, various conclusions could be drawn. The OS-1433 (Bakelite XV-1657) with a nitrocellulose sealer for the detent springs was better than many of the other unpigmented coatings tested. Vehicles showing equal or better promise were vinylite plasticized with either tricresyl phosphate or Aroclor 1254, Parlon-X (chlorinated synthetic rubber) plasticized with Aroclor 1254, and polyamide resin with Aroclor 5460. The Bakelite systems began to blister before two weeks. Vinylite and Parlon-X showed less corrosion and blistering, as did the polyamide resin. Butyl rubber and Saran coatings gave poorer results. Of the pigmented coatings the best was Bakelite XV-1657 pigmented with titanium oxide and talc, which showed no moisture penetration after 5 weeks and which does not require a nitrocellulose sealer for the detent springs. Zinc yellow pigmentation generally showed poorer results than titanium oxide and zinc oxide.

As a result of the laboratory and humidity cabinet tests three coatings were selected and recommended for surveillance tests on live ammunition. (1) Bakelite XV-1657 pigmented with titanium oxide, 27 per cent, and flake talc, 73 per cent by weight; (2) Vinylite VMCH (vinyl chloride-vinyl acetate copolymer modified with maleic anhydride) plasticized with Aroclor 1254 (chlorinated diphenyl liquid), and (3) pigmented Parlon-X plasticized with Aroclor 1254, pigmented as in (1).

17.4.3 Primers

The particular primer of concern was a delay element primer consisting of a copper primer cup, a layer of primer mixture on the bottom of the cup, and a tin sealing cup on top of the mixture. The space to be sealed was the annulus between the copper primer cup and the tin sealing cup. The sealant was dropped in after the two cups were assembled. The sealant previously used in production was shellac, which was used as the control in the tests.

Some thirty sealing compounds were prepared and tested for ability to seal the primer against air leakage at 40-cm water and for moisture transmission in vapors of 1 per cent ammonia solution at 140 F. For the latter tests the explosive mixture was replaced with dry silica gel, and the desiccant samples were weighed at intervals of two weeks. In addition to these laboratory tests one thousand loaded primers were subjected to a surveillance test at Frankford Arsenal. After storage the primers were fired, using a type of sensitivity test in which the height of fall of a firing weight is reported.

On the basis of the results obtained by the tests described, the sealant recommended was Parlon-X plasticized with tricresyl phosphate. This is considerably better than the shellac previously employed.

Chapter 18

CLEANING OF GASOLINE CONTAINERS FOR USE IN TRANSPORTING DRINKING WATER

18.1 SUMMARY

Gasoline containers may be cleaned for use in transporting drinking water by filling with water and activated carbon and agitating for a few minutes. The mixture consists of 1 pound of activated carbon in 50 gallons of water.

18.2 CLEANING METHODS FOR CONTAINERS

Since in emergencies it is often necessary to use gasoline containers for transportation and storage of drinking water, an investigation was made of methods of cleaning the gasoline containers and of purifying the water, so as to avoid danger from the toxicity of the gasoline or lead contained in it.

Several adsorbents were tested for their ability to remove color, odor, and lead from water contaminated with gasoline. These materials, in the order of their effectiveness, were: activated carbon, Amberlite IR-100, Zeo Karb, activated alumina, Amberlite IR-4, fuller's earth, Supercell, and sand.

The activated carbon could be used in the granular state in filter beds or added directly to the water in the powdered form and then filtered out by any available sand filter or strained out of small quantities of water by flannel cloth. For filter beds of 2-ft depth satisfactory results were obtained at a rate of filtering of 2.5 gallons per minute per sq ft area. If powdered carbon is added directly to the water, the amount required is 1 pound to 50 gallons of water, and the container should be agitated for 10 to 15 minutes.

The carbon treatment will rarely be necessary if the containers have been properly cleaned and if storage of water in the containers does not exceed 40 hours. Five-gallon tins can be cleaned effectively by a thorough rinse with soap and water, followed by several rinses with clear water. Fifty-gallon drums can best be cleaned by steaming for 30 minutes and then thoroughly rinsing with water to which a small amount of powdered carbon has been added. Gasoline tank trucks should be steamed for at least 90 minutes and thoroughly flushed with water to remove loose scale. When removed, the water should be filtered through sand filters, and added protection may be provided by the addition of 0.5 gram of powdered activated carbon for each gallon of water before the sand filtration. Gasoline tank cars should be cleaned in the same manner as the tank trucks except that the steaming should be for at least 6 hours.

Chapter 19

PRODUCTION OF MAGNESIUM FLUORIDE

19.1 SUMMARY

MAGNESIUM FLUORIDE is used to coat optical lenses to reduce reflection from the glass surfaces. Although little of the material was required, it was specially prepared by a complicated technique, because spattering and outgassing during the coating process caused the rejection of many lenses. In the investigation described it was found that a satisfactory material for the coating operation could be prepared by the simple process of melting impure commercial magnesium fluoride and holding it at 1475 C in a graphite crucible for 15 minutes.

19.2 PREPARATION OF FLUORIDE FOR COATING LENSES

Magnesium fluoride is used for coating military lenses, because it greatly decreases the light reflected from the glass surface and thereby increases the light transmitted. The coating operation is done in a bell jar under a very high vacuum, with the heated lenses held in the top of the jar. The magnesium fluoride, which is vaporized from a crucible in the bottom of the jar by a heated coil of tungsten wire close to the surface of the fluoride, condenses on the surface of the lenses. The apparent color of the lenses changes through the spectrum and the coating operation is stopped when the desired color (pink) is reached.

The standard Navy method of preparing magnesium fluoride was to heat chemically pure magnesium chloride with chemically pure hydrofluoric acid in a platinum crucible. Hydrofluoric acid was added as many times as necessary to remove all the chloride. The fluoride, after drying, was heated for several hours at 1050 C. This was a laborious and time-consuming operation which resulted in an expensive product. The material thus prepared occasionally sputtered and outgassed during the coating operation, causing the formation of soft films on the lenses as well as defects wherever solid particles struck the glass. An imperfect coating required regrinding of the lens, so that one of the serious costs of the process was the spoiled lenses rather than the high cost of the magnesium fluoride.

Experimental batches of magnesium fluoride were made from a variety of starting materials and given the standard heat treatment. While a fair product was obtained from most of the procedures, none of the products was definitely better than that made according to the Navy method. The various methods of preparation were:

1. Magnesium metal treated with hydrofluoric acid (resulted only in a slight reaction).
2. Magnesium metal treated with a mixture of nitric and hydrofluoric acids.
3. Magnesium metal treated with formic acid. Resulting magnesium formate converted to fluoride with hydrofluoric acid.
4. Magnesium metal treated with acetic acid. Resulting magnesium acetate converted to fluoride with hydrofluoric acid.
5. Magnesium metal burned in air. Resulting oxide and nitrite treated with hydrofluoric acid. (Burning of the metal was too vigorous for safety.)
6. Chemically pure magnesium nitrate treated with chemically pure hydrofluoric acid.
7. Magnesium fluosilicate decomposed to the fluoride by heating. (High cost and low yield.)
8. Chemically pure light magnesium oxide and dense magnesium oxide, commercial magnesium oxide from mineral magnesite, sea water oxide (light and dense), U.S.P. oxide (light and dense), changed to fluoride with both technical and chemically pure hydrofluoric acid.

Because of the similar, and unsatisfactory, results from all these samples, it was concluded that purity and method of production were not important factors, but that the heat treatment might be critical. It was suggested that if the fluoride were heated to fusion, most of the sputtering would be eliminated. The melting point of magnesium fluoride is 1395 C, which is higher than can be reached in an ordinary electrical or gas muffle furnace. An Ajax 3P2 induction furnace was therefore used for heating the fluoride to 1475 C. The melting required 5 to 8 minutes; sputtering occurred until the sample was well melted. Sputtering and outgassing of samples treated in this way were negligible during the coating operation.

Crucibles made from Acheson graphite were used for the fusion. Magnesium fluoride, at high temperatures, has exceptionally strong slagging characteristics. Clay, porcelain, alundum, Norbide, and magnesium oxide crucibles were rapidly destroyed. Platinum is seriously injured by contact with many materials at very high temperatures. The graphite at these temperatures oxidized, so that the more exposed top part of the crucible burned off and its capacity was gradually reduced until a new crucible had to be used. Some protection was obtained by covering the crucible with an inverted Norbide crucible.

Satisfactory samples of magnesium fluoride were obtained by fusion of material from every source. Since the purity did not appear to be important, commercial magnesium fluoride, selling for $17.00 per 100 pounds, was obtained from the Pennsylvania Salt Manufacturing Co. After this material had been held at a temperature of 1475 C for 15 minutes, cooled and ground, it was tested by the Navy and reported as satisfactory.

The analysis of this commercial fluoride was:

	Per Cent
Loss at 100 C	3.2
Loss on ignition	7.26
Water soluble	7.49
Silica	None
Calcium	None
Magnesium	34.40
Magnesium calculated as magnesium fluoride	88.06
Fluorine	54.0
Fluorine calculated as magnesium fluoride	88.55

Iron, which is a common impurity in these commercial samples of magnesium fluoride, is gradually reduced to metallic iron which collects as pellets in the bottom of the crucible. The longer the mass is kept molten, the more completely is the iron reduced, as shown by the fuzed mass becoming more white and less yellow in color. If the molten mass is allowed to cool and solidify in the crucible, a considerable portion of these pellets can be mechanically loosened and discarded. If the molten mass is poured from the crucible into a mold, these iron pellets become mixed with the magnesium fluoride and cannot be readily removed. There was no indication in any of the tests that the iron which was present in the final material caused any trouble. Molten magnesium fluoride contracts when it solidifies; hence, the solid magnesium fluoride can be easily removed from a crucible or mold.

The time that the material was held molten varied from a few minutes to 50 minutes and there was no apparent correlation with the results obtained. Fifteen minutes appeared to be adequate.

It was concluded that fusion of the magnesium fluoride before use was desirable regardless of the method of preparation, and that satisfactory material could be prepared by the simple fusion of commercial magnesium fluoride, thereby avoiding the troublesome standard method of preparation.

Chapter 20

SUBSTITUTE FOR CORK IN ORDNANCE PLUGS

20.1 SUMMARY

SATISFACTORY ORDNANCE plugs for shell casings can be made from waste wool shearings and a thermosetting phenolic resin, the molded product being coated with a beeswax-turpentine-graphite coating.

20.2 PLUGS FROM WOOL WASTE

Because of the shortage of cork the Navy Bureau of Ordnance desired the development of a substitute material which could be used for making plugs for shell casings. This material was to be made from noncritical materials; have approximately the same resilience, compressive strength, and aging characteristics as cork; afford similar protection against moisture; disintegrate upon firing; and be in line with cork in cost.

Materials considered were cellulosic agricultural wastes, paper pulp, leather scrap, animal hair, textile wastes, and reworked rags or shoddy, using binders such as pitch or asphalt, adhesives in water or other solvents, thermoplastic resins or thermosetting resins. The best of these proved to be wool shearings bound with about 20 per cent thermosetting phenolic resin. A waterproof coating of rubber latex or shellac was used, on top of which was painted the standard beeswax-turpentine-graphite coating specified for cork plugs.

The manufacture involves the following main steps.

1. Disintegrating and blending of the incoming wool stock.
2. Mixing molding powder and wool.
3. Weighing out and filling molds.
4. Molding or curing under heat.
5. Waterproof and beeswax coating.

The curing time in the mold was about 30 minutes at a temperature corresponding to a steam pressure of 110 pounds. Plugs for tests were made in a three-piece steel mold dimensioned to compensate for shrinkage. The mold was placed between the steam-heated platens of a hydraulic press with top and bottom surfaces in contact with the platens in order to transmit the heat necessary for curing. Plugs made in this manner satisfactorily fulfill the requirements.

Chapter 21

IMPROVEMENT OF SUBMARINE STORAGE BATTERIES

21.1 SUMMARY

An exploratory study indicated that the capacity of submarine storage batteries may be increased appreciably by employing stronger acid with appropriate modifications of the plate thicknesses and spacing.

21.2 USE OF STRONGER ACID

The ability of a submarine to remain underwater is limited by the capacity of its storage batteries. When these are discharged, it is necessary for the submarine to surface and recharge the batteries with the diesel engines. Since the batteries are exceedingly heavy, the number of them cannot be increased. If the capacity could be increased, the time under water could be increased or the weight carried could be reduced. Because of this situation, an exploratory study was made of possible methods of increasing the capacity of lead storage batteries.

The chemical reactions occurring in this type of battery are:

1. At the negative plate

$$2(+) + Pb + H_2SO_4 = PbSO_4 + 2H^+$$

2. At the positive plate

$$PbO_2 + H_2SO_4 + 2H^+ \rightarrow PbSO_4 + 2H_2O + 2(+)$$

From these equations, it is apparent that in the standard battery there is an excess of lead and lead oxide over that which will react with the acid. Theoretically, an increase in the concentration of the acid from the standard density of 1.28 to 1.40 would increase the capacity per unit volume by 45 per cent. Actually, however, the effect of strong acid is to decrease the capacity of the negative plate. This decrease in capacity occurs during discharge and is probably due to coating over a portion of the active lead of the negative plate with lead sulfate. The lead sulfate is less soluble in strong acid, and the strong acid is more viscous and so decreases the rate of diffusion. The positive plate is not injured by acid densities up to 1.50.

In the standard batteries the ratio of positive to negative plate thickness is 0.081 to 0.071 in. This ratio gives about equal utilization of positive and negative material using 1.28-density acid. When stronger acid is used, however, this ratio is not correct because the positive plate becomes relatively more efficient, and a decrease in the capacity of the battery results. However, if the plate thicknesses are adjusted to the proper ratio for the concentration of the acid used, a gain in the capacity of the battery can be obtained. The plates must be thickened and the acid space made less if full advantage is to be taken of use of stronger acid.

It was considered unwise to use acid stronger than 1.37 because the gain in capacity above that point involves use of exceedingly heavy batteries. Calculations based on experimental results indicated that by proper design it is possible to gain about 35 per cent increase in capacity for the same size battery by increasing the acid strength to 1.37, and changing the size of the plates and acid space. Such a battery would have negative plate thickness of 0.102 in., positive plate thickness of 0.101 in., and acid space of 0.045 in., as compared with the standard negative plate thickness of 0.071, positive plate thickness of 0.081, and acid space of 0.070. These calculations were checked roughly[1] by partially filling the acid space of standard cells with sand, and testing with the stronger acid. This battery showed an increase of capacity of 25 per cent. These experiments were carried no further, and should be checked, but they indicate that by suitable modification of plate thickness, spacing, and acid density, it may be possible to increase the capacity of submarine batteries by as much as 35 per cent.

Chapter 22

REMOVAL OF OIL FROM HARBOR WATERS

22.1 SUMMARY

OIL SPILLS may be removed from harbors by spreading "doped" sand on the water and carrying the oil to the bottom with the sand. The most effective dope was a mixture of higher aliphatic amines. The amount of dope and sand required is so large that the method is practical only in special situations.

Information on industrial techniques of handling oil spills was collected and reported.

22.2 USE OF TREATED SAND

Oil spills on harbor waters are apt to occur as a result of enemy action against ships and shore storage tanks. These spills are a hazard because of their inflammability, and the heavier oils are a serious nuisance to salvage operations. When the oil is in large patches on fairly unconfined water, the easiest and most economical method of collecting it is by skimming with suitable equipment. The available information on this method of removal was compiled.[1]

An investigation was made of the use of chemically doped sand for sinking the oil. Tests showed that thoroughly dried sand carries down the oil but does not hold it down. If the sand was treated with one of several effective chemicals, the amount of oil carried down was not increased but the oil was held on the bottom. The sand was treated simply by soaking in a solution of the chemical for a few minutes and draining. It was not necessary to dry it. The materials found effective for the treatment of the sand were Armour amine acetate, Armour-free amine, Shell paramine hydrochloride, and Tretolite Reagent L-28,097. All these are high molecular weight amines or the esters. Of these the Armour amine acetate had the optimum oil-sinking performance. This is the mixed acetates of 25 per cent mono-*n*-octadecyl amine ($CH_3(CH_2)_{16}CH_2NH_2$), 25 per cent mono-*n*-hexadecyl amine ($CH_3(CH_2)_{14}CH_2NH_2$), and 50 per cent mono-*n*-octadecenyl amine ($CH_3(CH_2)_7CH{:}CH(CH_2)_7CH_2NH_2$).

The ratio of the weight of oil carried down to weight of sand increased as the sand size decreased, but the amount of dope required also increased. Silica sands from many sources appeared to have comparable oil-sinking performance. Crushed limestone required more dope than the corresponding size of silica sand and was not so effective. The weight of sand and Armour amine acetate dope required per 1,000 barrels of oil for film reduction from 0.20 to 0.05 in. is given in the following table.

	Silica Sand Size (Inches)					
	0.0013		0.003		0.005	
	Sand (tons)	Dope (lb)	Sand	Dope	Sand	Dope
Gasoline	263	10,230	317	5,390	395	4,100
Diesel fuel	465	18,100	512	8,810	574	5,970
Bunker "C"	530	20,700	585	10,050	656	6,820

The sand-doping and oil-sinking processes were carried out in sea or fresh water with equal effectiveness.

A basic characteristic of the chemical-sand method is the fact that the oil cannot be reclaimed at reasonable cost after adsorption by the doped sand. This loss, in addition to the cost of the chemical (about 30 cents per pound), makes this procedure less economical than mechanical removal, but it may be more advantageous in special situations such as burning films, restricted areas such as dry docks, application by air guns already available, and so forth.

Chapter 23

PRODUCTION OF NITRIC ACID FROM URINE

23.1 SUMMARY

PRODUCTION OF NITRATES from urine is practical as an emergency measure in thickly populated countries. A solution of approximately 1 per cent ammonia may be obtained by the action of common bacteria on human urine, or by the use of the enzyme urease obtained by water extraction of soybean meal. One ton of ammonia is obtainable each day from the urine of about 85,000 people. The ammonia so obtained must be concentrated and oxidized to nitric acid in standard equipment.

Alternatively, the urine may be nitrified by natural bacteria in aerated beds of broken limestone or other material, producing from 1 cu m of bed about 30 grams of nitrate nitrogen per day as a solution containing 1.75 per cent calcium nitrate. This method obviates the special chemical plant equipment for ammonia oxidation.

23.2 INTRODUCTION

The loss of the Burma Road in 1942 cut off practically all supplies to China, so that every effort was made by the Chinese to increase their limited production facilities. One of their greatest needs was for propellent explosives for small arms ammunition. This use required nitrocellulose, but they had no adequate nitric acid production and their stock pile of nitrates (Chile saltpeter) was practically exhausted. One large source of nitrogen was human urine, and an investigation was made to determine the best way of utilizing this. The use of urine was deemed feasible because collection systems were in operation in most Chinese cities. No sewage systems were used and the urine was collected and spread on the farm lands as fertilizer. One ton of ammonia can be produced from about 35,000 gallons of urine, which is approximately the amount per day from 85,000 people. One ton of ammonia is sufficient to produce over 3 tons of 100 per cent nitric acid.

The nitrogen in human urine can be easily converted to ammonia by either bacterial or enzyme action. The product contains 0.8 to 1.0 per cent ammonia and must be concentrated by simple distillation to obtain a product suitable as feed to an ammonia oxidation plant for producing nitric acid. The Chinese contracted directly with a United States company for the design of an oxidation plant so that this part of the process was not investigated by NDRC. It is also possible to convert the nitrogen to nitrates completely by bacterial action. The results indicated that these processes were operable and that they might be practical where it was impossible to obtain equipment for a more modern process, and particularly where a collection system was already in operation. The choice of the process used would depend upon local conditions and supplies.

23.3 AMMONIFICATION[1]

A wide variety of bacteria, actinomyces, and fungi split urea to form ammonia and carbon dioxide. These occur in soils, fresh and salt waters, muds, sewage, and the feces of man and animals. They abound in manured soils. Vigorous ammonifiers appear in practically all genera of saprophytic bacteria, cocci, and bacilli. The process goes on under both aerobic and anaerobic conditions and over broad ranges of temperature and hydrogen-ion concentration.

Normally voided urine, collected without sterile precautions and stored without adding bacteriostatic preservatives, will be naturally inoculated with a mixed culture of urea-splitting organisms and others capable of hydrolyzing uric and hippuric acids almost quantitatively to ammonia. The practical problems in large-scale processes are (1) bringing about ammonification in a convenient interval, and (2) preventing loss of volatile ammonia.

Conversion of the urea to ammonia can be accomplished by simply letting it stand, but this may require as long as a week. If inoculated with 10 per cent of fermented urine from a previous batch, the reaction will be completed in about 24 hours. Because of the ease of this reaction, ammonia will be formed before and during collections, and precautions should be taken to prevent loss of this ammonia. Such precautions are frequent collections (each day preferably) and the use of covered containers, or containers having a minimum surface exposed to the air.

A series of tanks should be provided for fermentation. The size of the tanks is not important and may be governed by the ease of fabrication and the necessity of heating them. They may be made of any material capable of resisting 1 per cent ammonia at *p*H 9.0. Either iron or ceramic is satisfactory, the iron, of course, being the easier to heat. Since the loss of ammonia occurs at the air-liquid interface, this area should be kept as small as possible by using covered deep tanks. Some means of heating the tanks slightly must be provided. Direct firing should be satisfactory, or pre-heated urine may be run into wood vats or tanks kept in a warm room.

Human urine, the urine of domestic animals, and the feces of domestic fowls may be used. Pollution with animal or human feces, rubbish, paper, etc., however, will actually cause a lower yield by binding part of the ammonia nitrogen. The material is placed in the tanks with 10 per cent of the material left from the previous batch and heated, if necessary, to bring the temperature to 25 to 30 C. At 20 C, or below the rate of ammonification, the process is not only slower, but is also unpredictable and varies more from one batch to another than at the higher temperature. The first batch may be started by seeding with old urine, manure, or soil. When the ammonification is complete, 90 per cent of the fermented urine should be drawn off and replaced with new urine. The remaining 10 per cent of each batch should be an adequate starter for the new charge. For a batch seeded with 10 per cent of fermented urine, the hydrolysis to ammonia should be complete in about 24 hours.

There will be no effervescence during the fermentation unless gross organic pollution of the urine occurs, and only the volume changes due to temperature variation of the liquid need to be considered when estimating the space to be left at the top of the tank.

The completion of the hydrolysis may be determined approximately by titrating with standard acid using methyl red as indicator. When no further increase in the titration is observed, the hydrolysis is essentially complete.

The effect of dilution on the reaction was investigated and it was found that a 50 per cent dilution speeded up ammonification in batches without inoculation, but was of no advantage if inoculation was used. Dilution was, of course, a disadvantage because of greater difficulty of concentrating.

Under aerobic rather than anaerobic conditions, the ammonification proceeded at about the same rate but the loss of ammonia was excessive, often amounting to more than 50 per cent of the total ammonia. If the process is carried out in deep tanks, the system becomes anaerobic within a few hours because of the biological oxidation of the reducing sugar in the urine, and this condition of operation was recommended.

The *p*H over the range encountered did not inhibit the bacterial action. No accessory nutrients were necessary for the bacteria.

The nitrogen content of urine can also be converted to ammonia by the enzyme urease instead of by bacteria. This has the advantage of requiring less time for the completion of the reaction and the disadvantages of being inhibited by a high *p*H and requiring dilution by 10 per cent additional water.

Soybeans contain urease which can be extracted by mixing one part of soybean meal with ten parts of water for ½ to 1 hour with occasional agitation, and finally allowing the mixture to settle and drawing off the top liquid. The fineness of the bean meal is not important; roughly 20-mesh or finer is satisfactory. The meal which is left is not harmed and may be used as food. The mixing tanks preferably should be of ceramics or wood, since the salts of many metals have an inhibiting effect on the urease. No heating is necessary and the required agitation can be done with hand paddles if desired. The urease extract must be used within 48 hours from the time it is prepared and preferably the same day.

One part of the urease extract is added to ten parts of urine and the temperature raised to about 30 C for best results. A higher temperature results in a faster reaction but also in greater loss of ammonia. The urease becomes inactive at about 60 C.

The urease becomes unreactive if the *p*H of the urine rises to 8.8. By the time the alkalinity reaches this figure the reaction should be practically complete. If for any reason it is not, the alkalinity can be reduced by bubbling carbon dioxide through the liquid. In fact, the optimum *p*H for the reaction is 7, and if carbon dioxide is available, it is an excellent material for controlling the alkalinity. By bubbling carbon dioxide through the mixture for 5 minutes at approximately 30-minute intervals, the time required for the conversion will be reduced to about three quarters of that otherwise required.

The enzyme process proceeds more rapidly when the *p*H of the solution is below 8. The bacterial process works faster at a *p*H above 8. A combination of the two processes allows each to operate at its preferred *p*H and results in a faster reaction than when either reaction is used alone. The bacterial process is started exactly as the enzyme process by adding one part of urease extract to ten parts of urine. If the temperature is kept at about 30 C, the ammonification will be about 40 per cent complete in 1 hour and the *p*H will have risen to above 8. At this point, 10 per cent of fermented urine from a previous batch is added and the reaction allowed to go to completion.

The approximate times for complete ammonification by these processes are:

	38 C	28 C
Bacterial process	12 hr	24 hr
Enzyme process	4	8
Combination	2	4

The ammonia solution obtained by either bacterial or urease treatment of urine will contain about 1 per cent ammonia. This must be concentrated for subsequent use in making nitric acid or ammonium salts. A laboratory investigation of the stripping of converted urine was carried out in order to check the simple ammonia-water distillation calculations and to obtain some experience with regard to the foaming problem. This work showed that the standard ammonia-water equilibrium data can be used to calculate the result of distillation of ammonia from the fermented urine. Addition of lime to the fermented urine before distillation slightly increased the yield of ammonia; it seemed to have little effect on foaming.

A plant design was worked out for the concentration, which was unique in that construction materials were limited to nonmetallic materials, since metals were extremely scarce in China.[1]

23.4 BIOLOGICAL NITRIFICATION OF URINE[2]

The nitrogen in human urine may be converted to nitrates by subjecting it to the action of a mixed natural flora of nitrifying bacteria upon beds of crushed limestone coated with small amounts of soil. About 30 grams of nitrate nitrogen may be obtained per cubic meter of bed per day. The effluent is a 1.75 per cent solution of calcium nitrate together with other urine salts. Efficiencies depend upon care of operation; under usual conditions about 75 per cent recovery may be expected; but with adequate laboratory control this may be improved to 90 per cent or better.

Nitrates are produced naturally in many soil and water systems. The conversion of ammonia to nitrate by bacteria takes place in two recognizable steps: (1) the oxidation of ammonia to nitrite by species of Nitrosomonas and Nitrosococcus, and (2) the further oxidation of nitrite to nitrate by members of the genus Nitrobacter. These organisms occur in practically all natural media and are particularly abundant in the aerated, surface layers of fertilized soils, on the upper few inches of slow sand sewage filters, and in shallow, lightly polluted surface waters.

Previous methods of intensive nitrification of ammonia were reviewed and tested along with other promising processes. Some of the methods previously recomended for ammonia did not apply to urine because of its limited nitrogen content, its chloride, carbonate, sulfate, and phosphate content, its alkaline reaction after fermentation, and its high surface activity.

The conditions for optimum activity of individual species in pure culture vary widely, but in natural systems where a number of species are simultaneously active the following appear to be most favorable.

1. A large surface area such as may be provided by loose straw or peat beds, by cinders, charcoal crumbs, crushed rock, coarse sand, or crumbly soil.
2. A supply of oxygen furnished by moving liquids or by diffusion of air into films of liquid on wetted surfaces; anaerobic conditions block nitrification.
3. Moisture sufficient to maintain a film of water and nutrients over the slime of bacteria attached to surfaces of the solid medium.
4. Moderate temperatures: systems adapt themselves to the prevailing temperatures, but nitrification appears to take place slowly at temperatures close to freezing; the optimum range of temperatures lies between 25 C and 30 C.
5. The presence of bicarbonates: nitrifying bacteria are autotrophic in their metabolism and utilize this source of carbon for cell synthesis.
6. The presence of calcium or magnesium base in amounts slightly in excess of that required to combine with the nitric acid formed. Porous limestone itself forms an excellent surface for the activity of nitrifiers.
7. Favorable hydrogen-ion concentrations usually lying on the alkaline side of neutrality but varying with the condition of the system. Nitrification usually ceases at *p*H values below 4.0 and above 10.3.
8. Nitrifying systems with ammonium concentration maintained at levels below 1,000 ppm nitrogen. Activity usually begins earlier if lower concentrations are used. It is possible to raise the concentration as the system develops, and in some organic-rich soils concentrations in excess of 1,000 ppm nitrogen may be nitrified.
9. A system free of readily decomposed organic matter. The presence of considerable quantities of soluble and readily available organic matter makes possible the growth of competitive organisms. The metabolism of the nonnitrifying flora may produce anaerobic conditions and bring about the reduction of preformed nitrates and their loss as nitrogen gas. For this reason it is desirable that support material be inert to bacterial activity.

In the system recommended, dilute urine was percolated over beds of ½-in. limestone, lightly coated with activated nitrifying soil. After nitrification had begun the concentration of nitrogen in the system was built to an optimum level by adding ammonified urine to the effluent and recirculating the mixture over the beds. Chemical control of the system was obtained by observing the concentration of nitrite. Good conditions are indicated by traces or total absence of nitrite, unsatisfactory conditions by rising nitrite concentrations. This measurement provided a convenient indication for increasing or decreasing the rate of feeding urine.

From the performance of experimental beds, it was found that approximately 0.3 cu m of crushed limestone per capita was required. This assumed that the average nitrogen yield of urine lay between 8 and 12 grams per day per person. Beds 0.5 m deep were used, but deeper beds may be used. The practical limit is determined by the accumulation of silt washed from the upper layers of stone into grids and lower layers of the bed. Ventilation is adequate in beds 1.0 m deep and possibly deeper if existing soil is removed. Small limestone lumps gave the best results because they presented the maximum active surface. If finer than ¼ in. to ½ in., however, they tended to clog after a few weeks' operation and ventilation problems developed. The quantity of nitrifying soil used varied with the soil type, but represented approximately 5 per cent by weight. The proportion of soil must be determined empirically by finding how much will adhere to samples under conditions of recirculation. It is advisable to prepare the nitrifying soil as soon as operations can be started, since about six to eight weeks will be required to obtain an active soil.

Beds should rest upon false bottoms to permit rapid draining and to prevent the formation of pools. If stagnant zones developed, denitrification and loss of nitrogen occurred. A further advantage was the better ventilation of beds resting on grid work or other false bottoms. Such beds are less likely to become waterlogged by rain storms. The effluent of the beds was recirculated at rates of 150 to 300 liters per cubic meter per day, and ammonified urine was added to the recirculating system at rates of 4 to 6 liters per cubic meter per day. Reservoir bottoms were kept small so that as much of the liquid as possible would be on the beds. Practically, this depended upon rates of evaporation and the frequency with which the volumes were replenished—a reservoir volume of approximately 40 liters per cubic meter of bed was adequate.

The beds may be operated as continuous systems. In order to obtain ammonia-free effluents, however, a period of recirculation without added urine must be allowed. It was recommended that the bed be fed urine through the day and allowed to oxidize the final additions of ammonia during 6 to 8 hours of the night, and that the unneeded nitrified effluent be drawn off in the morning before feeding is resumed.

If possible, the plant should be sheltered with a raintight roof. The bed will be less subject to diurnal fluctuations in temperatures and evaporation losses will be reduced. No damage to the nitrified flora occurs from rains or temporary flooding of the beds beyond the dilution of the effluent. The bacteria will retain their activity. Drying of the bed, however, is followed by a drop in activity extending over a week or more. Should this occur, it is not necessary to remake the bed with fresh earth, as the dried bed will gain its full activity in a shorter time than new beds required to come to full activity.

Fields for solar evaporation or equipment must be provided for evaporating approximately 3 liters of effluent per person per day.

[redacted]

Chapter 24

SUPPRESSION OF DUST AROUND ARTILLERY EMPLACEMENTS

24.1 INTRODUCTION

The firing of field artillery pieces over dry ground raises a considerable cloud of dust, which is a distinct disadvantage because it obscures the gunner's vision so that he cannot observe the impact of the projectile and correct his aim in succeeding rounds, and because it reveals the gun position to enemy observers. This condition is aggravated by the practice of digging in the fieldpiece in order to present a lower silhouette. Often the muzzle of the gun is only 1 to 2 ft above the ground. This digging in results in loose dirt around the gun and by placing the muzzle near the ground greatly increases the blast on the dirt.

An investigation of methods of suppressing the dust was carried out along two lines: (1) a chemical treatment that could be applied readily to the dust and which would bind it to such an extent that it would not be disturbed by the blast, and (2) a portable mat of suitable size and material so that when laid on the ground in front of a gun it would withstand the blast and allay the dust.[a]

24.2 CHEMICAL TREATMENT OF GROUND[2]

A number of common chemicals that might be suitable for binding the loose surface dirt were tested. These chemicals included casein, sodium alginate, cane sugar molasses, corn syrup, calcium chloride, library paste, ordinary glue, linseed oil, sodium silicate, polyvinyl alcohol, clear automobile lacquer, a quick-set gypsum (Hydrostone). Water solutions or emulsions were considered to be most desirable, and wetting agents would be needed to gain penetration. Wetting agents suggested were: abietic acid, Aerosol OS and OT, Dreft, Nacconol F and NR. To form emulsions various peptizing media were suggested: monoethanolamine and triethanolamine, cream of tartar, sodium hydroxide.

Artificial soils composed of mixtures of finely divided clay (Drilloid, or drilling mud clay) and fine sand were prepared and placed in shallow glass baking dishes. An air gun was built to operate on compressed air at approximately 100 psi pressure, and fitted with a $\frac{3}{4}$-in. orifice and quick-opening valve. The surface of the artificial soil bound with a given chemical was blasted with the air gun. The binders were rated according to their ability to withstand the blast of the laboratory air gun and the most promising ones were tested on natural ground subjected to the muzzle blasts of 76-mm and 90-mm guns.

Water was first tried with the addition of only a wetting agent to obtain penetration (Aerosol OT in a concentration of 0.1 per cent was the best wetting agent for this use). This was found unsatisfactory unless impractically large amounts were applied.

The primary purpose of the deliquescent materials (black strap molasses, corn syrup, calcium chloride) was to retain moisture. As might be expected, aqueous solutions of these materials were little, if any, better in binding power than water alone.

These tests indicated that it was necessary to bind the upper surface of the ground to form a slab strong enough to resist the tension, compression, and shear stresses induced in it by the shock of the blast. A high endurance limit was essential.

Casein, library paste, and ordinary glue were tried, but found to give weak crusts that were very slow in attaining strength. They were no better than water with wetting agent, failing after a few tests from the laboratory gun. Polyvinyl alcohol was difficult to put into water solution at room temperature and was very slow-setting unless heat was applied. An attempt to speed the set by the addition of hydrogen peroxide showed no appreciable benefit. An emulsion of linseed oil and water was made with cobalt resinate added to speed the attainment of strength by the linseed oil. When sprayed on the soil, the emulsion broke down and failed to wet the dust. All the wetting agents were tried, but none was satisfactory either in obtaining penetration or stopping the breakdown of the emulsion.

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[a] Division 2 worked on this problem from the point of view of developing improved blast deflectors, which would disperse the blast sufficiently to materially reduce the dust raised.

Alginates (Keltex and Kelgum) gave fairly good results in the laboratory and were deemed worthy of full-scale tests.

Excellent laboratory results were obtained with sodium silicate, a 40 per cent clear lacquer with a 60 per cent lacquer thinner solution, and Hydrostone. Philadelphia Quartz Company D Brand sodium silicate was used, which contained about 44 per cent sodium silicate and 56 per cent water. Slight reduction of the water caused the liquid to solidify into a hard, very strong mass. Penetration was obtained by using 0.1 per cent Aerosol OT in the water used to dilute the sodium silicate as obtained from the manufacturer. It was found that a 1:1 solution by weight (1 part D Brand sodium silicate and 1 part 0.1 per cent Aerosol OT water solution) penetrated well, had excellent strength after being allowed to set 1 hour, and withstood the air-gun blasts well. One hundred blasts did not disturb the soils in the pans. Philadelphia Quartz Company GC Brand powder is the dehydrated form of D, and has the advantage of better transportability.

Various percentages of lacquer were tried in the laboratory. A volume mixture of 40 per cent lacquer and 60 per cent thinner was found to form a dense, hard crust about ¾ in. thick on a wide variety of sand-clay mixtures. One gallon of the mixture per square yard of treated surface appeared satisfactory. Repeated blastings up to 75 times with the air gun in the laboratory were withstood without visible damage to the soils. A disadvantage for this material was that about 8 hours were required from time of application until the material had dried sufficiently for testing.

Various gypsum plasters were added in varying amounts to artificial soils in the laboratory. Those tried were art plaster, dental plaster, moulding plaster, and Hydrostone. Of these the most promising was Hydrostone, and it was tested most thoroughly. The manufacturers (United States Gypsum Company) claimed a tensile strength of 850 psi and compressive strength of 11,000 psi for a normal consistency neat plaster paste (about 32 per cent water by weight). Such a plaster was too stiff to permit addition to soil, but increasing the water to 60 per cent by weight gave good workability and the plaster-soil mix was hard in 1 hour after application. In the laboratory, as small amounts of plaster as 10 per cent of the weight of the soil gave excellent resistance to air gun blasts.

In full-scale tests with a 76-mm tank destroyer gun at Camp Hood, Texas, the sodium alginates formed gums that were too weak to withstand blasts even from high muzzle heights, and were destroyed by the first round.

Sodium silicates formed crusts that withstood the muzzle blast fairly well. The Philadelphia D Brand silicate diluted with 1 part of water (to which was added 0.1 per cent Aerosol OT clear) when sprayed from a knapsack-type sprayer at the rate of 1 gallon of solution per square yard of ground surface formed an excellent crust 1 hour after application. The crust withstood many rounds from muzzle heights 4 ft to 7 ft above the treated soil, but was destroyed by 10 to 15 rounds at low muzzle height (15 in.)

It was concluded that sodium silicate would not form a sufficiently strong crust to withstand continued firing at low muzzle height even when muzzle brakes were used on a 76-mm gun, and would be totally unsatisfactory for use without muzzle brake or under rounds from larger guns. Sodium silicate seemed to possess sufficient merit as a binding agent to use it on areas of low blast intensity if a more feasible method could not be found for alaying dust.

Contrary to laboratory tests, the clear lacquer was of no advantage in laying dust of the natural soil treated at Camp Hood. The first round from a 76-mm gun with muzzle height of 17 in. and without muzzle brake completely destroyed the treated surface and raised great clouds of dust as though the soil had been untreated.

The Hydrostone was spread over the loose soil as uniformly as possible and raked in with a garden rake to a depth of 1 to 2 in.; then water containing 0.1 per cent Aerosol OT was poured on the mixture until an amount equal to 0.6 per cent of the weight of Hydrostone had been added. After 1 hour two rounds from a

[redacted]

3-in. gun without muzzle brake and at muzzle height of 15 in. were fired over the treated position. The first round cracked and broke the surface, the second destroyed it and raised large white dust clouds. The position used in the test was on a side hill slope (about 6 per cent). The water ran and collected in pools in the depressions leaving the summits with insufficient water to hydrate the plaster. The low places were strong and undisintegrated by the two rounds, but sufficient high spots were weak, chalky, and dusty underneath to make the method unsatisfactory.

These tests demonstrated that none of the tried chemical treatments resulted in crusts sufficiently strong to withstand blasts generated in direct fire from prepared gun positions whether or not muzzle brakes were used.

24.3 BLAST MATS[3]

Preliminary tests of cotton canvas mats 15 x 18 ft for dust suppression were made by the Tank Destroyer Board at Camp Hood, Texas. The canvas had a tensile strength of 345 pounds per in. width. These tests showed that the mats markedly reduced the amount of dust raised and decreased the time of obscuration of the target. The canvas mats, however, were not of sufficient strength to withstand continued firing at low muzzle heights with the 3-in. or 76-mm guns, with or without muzzle brake. A search was, therefore, made for a material which was strong enough for this use.

24.3.1 Nylon

Nylon cloth suggested promise because of the known high strength and light weight of Nylon. Because of its fairly low melting point, however, there was doubt as to the effect of the heat from the blast. Preliminary tests were made by sewing 38-in. wide strips of Nylon duck to a canvas mat and subjecting it to the blast from a 76-mm gun without muzzle brake, using a muzzle height of 15 in. This Nylon weighed about 10 oz per sq yd and had a tensile strength of 800 pounds per in. width. It carried the du Pont code number NFD-191/2. After ten rounds the seams were loosened and the Nylon somewhat scorched and glazed at the area of maximum blast. Further tests with a 3-in. gun at 10-in. muzzle height aggravated these effects indicating that the material was strong enough but that it was adversely affected by the heat. The tests emphasized the need for strong seams and grommeting, grommets being necessary for pegging the mat to the ground.

The Army Ordnance Department had a full-size (18 x 24 ft) mat made from a double thickness of the same Nylon duck.[1] Seams were lock-stitched with Nylon thread and the two thicknesses of cloth were quilted together every 6 in. Grommets were placed every 3 ft over the entire mat, as shown in Figure 1. Tests of this mat verified previous conclusions. The mat was strong enough until weakened by the heat, after which it tore. The effect of the heat was particularly severe when flashing ammunition was used at low bore heights.

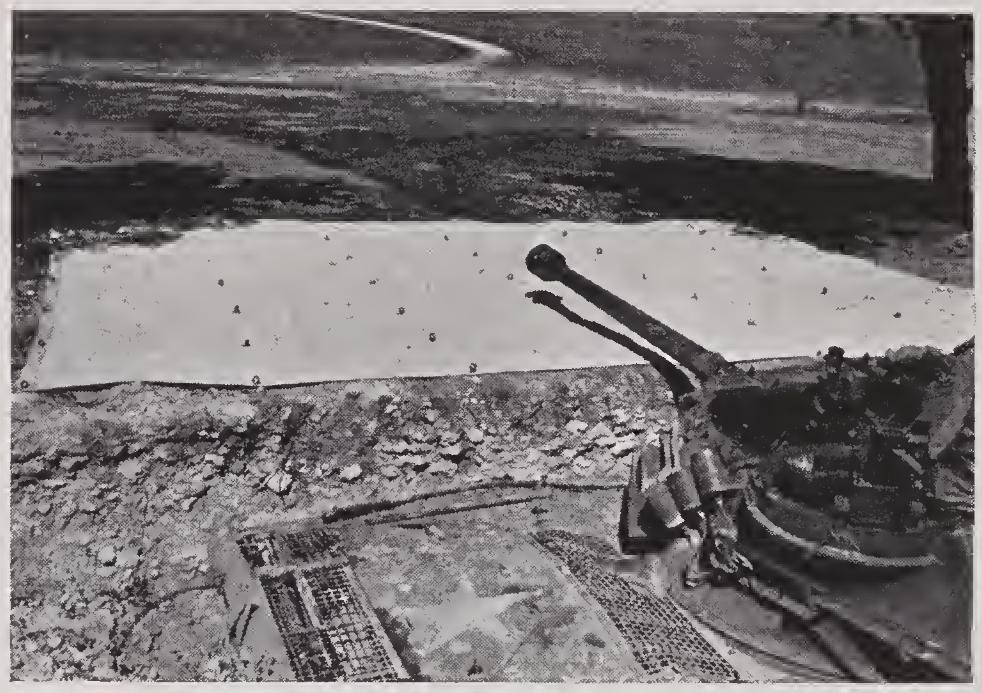

FIGURE 1. Nylon duck blast mat under 76-mm gun.

It was apparent that if advantage was to be taken of the good properties of the Nylon, some surface protection must be given to it. Two mats were therefore fabricated, one of a double thickness of the 19-oz Nylon, with the top surface coated with Vinylite; the other of a single thickness of 13-oz Nylon with the top surface coated with Perbunan synthetic rubber.

The Vinylite-coated Nylon was considerably better than the uncoated Nylon. When the 90-mm gun was used at a bore height of 12 in., however, the Vinylite coating was gradually removed until the Nylon was exposed and fuzed and the mat torn.

The Perbunan-coated Nylon was the best material tried. It withstood six rounds with the 76-mm gun and 14 rounds with the 90-mm, ten of the latter at a bore height of 12 in. The rubber satisfactorily protected the Nylon surface from the heat. Failure occurred from holes punched in the mat by stones underneath. The heavier Nylon, particularly if used in two thicknesses, would probably have given better results.

24.3.2 Vinylite

Preliminary tests with strips of sheet Vinylite sewed to canvas indicated the material was satisfactory if proper seams and grommets were used. This material was 0.08 in. thick, weighed about 4½ pounds per sq yd, and had a tensile strength of 110 pounds per in. width. Although the tensile strength was low, the elongation before break was 350 per cent, which resulted in a large part of the force of the blast being absorbed before the breaking point was reached. This material carried the Bakelite Corporation code number F.6988.

A full-size mat was made of this material except that the thickness was reduced to 0.06 in. in order to keep the weight within the desired 3½ pounds per sq yd. This material was not so good as the rubber-coated Nylon, but it was one of the best tried. In the tests it withstood 17 rounds of the 76-mm and 90-mm guns, but was destroyed by stone cuts when used with the 90-mm gun at a bore height of 15 in.

In order to determine whether there was any advantage to mats which allowed air to penetrate them, a mat was woven from 1-in. Vinylite strips $\frac{1}{16}$ in. thick. This mat, however, was too heavy (12 pounds per sq yd) and the woven surface apparently offered too much resistance to the blast. One round of the 90-mm gun at a bore height of 11 in. destroyed the mat.

24.3.3 Neoprene

Since Neoprene has greater tensile strength and abrasion resistance than Vinylite, a mat was made of sheet Neoprene 0.06 in. thick. The results, however, with this mat were not so good as with the Vinylite. The mat was destroyed with the 3-in. gun at a bore height of 12 in. The poor results may have been due to a rather rough surface, as opposed to a glossy, smooth surface on the Vinylite, which offered more resistance to the blast.

A mat made by sealing a loosely woven elastic cotton stockinet between two layers of Neoprene gave better results. The stockinet apparently furnished additional tear resistance to the Neoprene. Since the conditions of firing were not the same as for the Vinylite, a comparison of the two is difficult, but this mat was probably as good and would have given better results if it had had a smooth surface.

24.3.4 Fabrication

As stated earlier, the fabrication of the mat is as important as the material used. The first mats tested failed at the seams or grommets rather than in the material itself. The blast broke the thread of any exposed stitching, even when Nylon thread was used. To avoid this, cementing or heat sealing of the seams was used whenever possible. With fabrics, which required stitching, triple stitching with Nylon thread was used. Lock stitches were essential. All stitching was protected from the blast by covering it with a strip of Vinylite, rubber, or other suitable material.

Two types of grommets were found satisfactory. On the Vinylite mat the area of the grommet was reinforced by cementing on additional thicknesses of the mat material, and the whole clamped between two 6-in. diameter 16-gauge steel plates. The plates were held by a ¾-in. pipe nipple through the center of the plates with nuts on each end. On the two Neoprene mats two 6-in. diameter steel plates with ¾-in. center holes were placed on each side of the mat and held by six stove bolts ⅛-in. in diameter.

24.4 CONCLUSIONS

Blast mats are more satisfactory for suppressing dust around artillery emplacements than is chemical treatment of the ground. With the best chemical found, the preparation of a position required about 250 pounds of sodium silicate and 250 pounds of water. A sprayer was also necessary. A blast mat weighing less than 150 pounds can be used repeatedly in different positions and will withstand greater blast.

While the mats tested could not stand up under firing at very low bore heights, they very substantially reduced the dust and would be practical when bore heights above the minimum are used. Trials would have to be made to determine what height could be used continually with the best mats, but in all probability they would be satisfactory at bore heights of 18 in. or higher with the 76-mm gun and 21 to 24-in. or higher with the 90-mm gun. If improved blast deflectors are developed, the mats may be satisfactory at any muzzle height used.

It is estimated that for 3-in, 76-mm, and 90-mm guns blast mats should be about 18 x 24 ft.

Chapter 25

MANUFACTURE OF HYDROGEN PEROXIDE

25.1 SUMMARY

At the request of NDRC, an investigation of methods of production of concentrated hydrogen peroxide was undertaken. Shortly after the investigation was started, information on the German processes became available, and the program was curtailed. A general report on hydrogen peroxide technology was prepared.

Small-scale laboratory work showed the feasibility of producing exceptionally pure 60 to 65 per cent hydrogen peroxide by means of a glow discharge in water vapor at 0.15 to 0.32 mm Hg pressure. A condenser cooled with liquid air is required, and the power requirement is relatively high.

Hydrogen peroxide at strengths up to 90 per cent was obtained by a photochemical synthesis using ultraviolet radiation of a mixture of hydrogen and oxygen carrying a trace of mercury vapor. This process operated well at atmospheric temperature and pressure with a power consumption about twice that of the commercial electrochemical methods.

An investigation of the application of modern fluidized powder techniques to the old barium peroxide process for hydrogen peroxide indicated the possibility of a cyclic continuous process involving decomposition of barium nitrate, oxidation to the peroxide, reaction with acid, and recycling of the barium. Estimated power, fuel, and material costs for such a process are extremely low.

25.2 INTRODUCTION

One of the most important chemical developments by the enemy during World War II was the German use of concentrated hydrogen peroxide as a source of energy and oxygen in various new munitions. Peroxide was used as a rocket fuel in the Hs-293 glide bomb, in the Me-163 jet-propelled aircraft, and in various assist-take-off devices for aircraft. It was employed as a propellant in launching the V-1 "flying bombs," and as a source of power for a turbine to drive the fuel pumps of the German V-2 weapons. Wakeless long-range peroxide-driven torpedoes were under development at Kiel at the close of the war. Perhaps the most important development was a peroxide-driven submarine capable of 25 knots under water; the introduction of this submarine in large numbers might have disrupted shipping to England.

The interest of the United States in military applications of peroxide became keen in the winter of 1944-1945, at a time when supplies of commercial peroxide in this country were seriously short. Section 11.2 was asked in February 1945 to investigate methods of manufacture of concentrated peroxide and proceeded with an experimental program covering both manufacturing methods and means for concentrating dilute aqueous solutions. These studies were barely under way when the Rhineland was occupied and detailed reports of German manufacturing techniques began to come in. At about the same time, an American manufacturer (Buffalo Electrochemicals Co.) volunteered information concerning their development of a pilot plant for producing 90 per cent solutions of peroxide by a distillation process starting with commercial 30 per cent solutions, and an English concern (Messrs. Laporte, Ltd.) constructed a pilot plant of a similar type. Because of these developments the NDRC-sponsored program was not pushed, and only two small contracts were continued until the fall of 1945.

As a basis for planning the program contemplated in early 1945, a study was made of the possible methods of manufacture, the various military uses, and the existing technical information relating to hydrogen peroxide. This study was described in a general report[2] which was issued in the summer of 1945, and which may be referred to for a summary of the state of the art at that time.

25.3 PRODUCTION OF HYDROGEN PEROXIDE BY PHOTOCHEMICAL AND ELECTRIC DISCHARGE METHODS[4]

25.3.1 Introduction

At the time the German use of hydrogen peroxide was first disclosed, it was considered likely that the concentrated solution was obtained directly by a process involving electric discharge in water vapor at low pressure. Although the large-scale German production was later found to have been obtained by distillation of 30 per cent solution produced by electrolysis, a pilot plant based on the electric discharge method[2] was located at Elektrochemisches Werke München at Hollriegelskreuth and is described in intelligence reports. The Germans appear to have thought the process to be entirely practical for production of strong peroxide under conditions of high labor costs and low power costs. Before these facts were known, NDRC had initiated an exploratory study of the electric discharge and related methods.

25.3.2 Glow Discharge in Water Vapor

The glow discharge apparatus consisted of a 25-mm glass tube 176 cm between aluminum electrodes. Water vapor was introduced through a controlled capillary leak and traversed 35 cm of the discharge tube before being drawn off through a liquid air trap. Pressure was maintained in the range 0.1 to 0.6 mm Hg. The primary of the transformer was supplied with 60-c 110-v alternating current, and the secondary leads connected directly to the discharge tube electrodes, with a maximum secondary voltage of 14,000. The amount of water vapor fed was measured, and the condensate obtained in the liquid air trap was weighed and analyzed for hydrogen peroxide.

Optimum results were obtained with a steady full discharge, rosy pink in color, with a power input to the primary of 300 to 380 w, a secondary voltage of 600 to 650, and at pressures of 0.15 to 0.32 mm Hg. The condensate obtained consisted of 60 to 65 per cent hydrogen peroxide and 35 to 40 per cent water.

Complex ionization processes occur in the glow discharge resulting in uncharged atomic hydrogen and hydroxyl radical. Losses are believed to be due to combination of hydrogen atoms on the walls of the tube to produce molecular hydrogen, and to recombination of hydrogen atoms and hydroxyl radicals to form water. Hydrogen peroxide is presumably formed by direct combination of hydroxyl radicals:

$$OH + OH \rightarrow H_2O_2$$

Unless surface is provided at a temperature about that of liquid air the hydroxyl radicals do not condense and no peroxide is formed. This is believed to be due to the volatility of hydroxyl radicals, which is probably about that of hydrogen chloride. Condensation must occur promptly or the hydroxyl radicals react in the vapor phase to produce molecular hydrogen and oxygen directly. It is necessary to have both rapid condensation and cooling to about liquid air temperatures; rapid cooling with a solid carbon dioxide trap gives no peroxide product. The addition of oxygen to the water vapor feed did not improve the peroxide yield, as had been thought possible due to reaction of oxygen with hydrogen atoms. If it is assumed that a hydroxyl radical has the same chance of combining with another hydroxyl radical to form peroxide as it has of reacting with a hydrogen atom to form water, then the product should consist of an equimolal mixture of water and peroxide, containing 65 per cent hydrogen peroxide by weight. This checks the observed maximum concentrations of 60 to 65 per cent.

With the laboratory equipment employed the power consumed was very large in relation to the amount of peroxide formed. It is estimated, however, that only about 10 per cent of the power input was actually expended in the dissociation of water vapor. On this basis the experimental result of 0.013 gram of 55 per cent peroxide solution per minute with a total power input of 330 watts corresponds to 35 kilowatt-hours per pound 100 per cent hydrogen peroxide. This compares with 28 kilowatt-hours per pound reported by the Germans[2] for their electric discharge process, and 6 to 8 kilowatt-hours per pound for the commercial electrochemical processes. It is estimated that the theoretical minimum for the glow discharge process is about 3.6 kilowatt-hours per pound.

The glow discharge process has the advantages of requiring only water as raw material and of producing a very pure product at relatively high concentrations. The disadvantages are the relatively high power requirements and the low pressure at which the operation must be carried out.

25.3.3 Photochemical Synthesis

The photochemical process involves direct synthesis from hydrogen and oxygen at atmospheric temperature and pressure. The reaction is accomplished by illuminating the mixed gases containing a trace of mercury vapor with ultraviolet light of wavelength 2,537 A. The gases are saturated with mercury vapor at room temperature, the resulting mercury concentration being sufficient to absorb almost all the incident radiation in a light path of a few millimeters. The light quanta absorbed cause the formation of activated mercury which reacts with hydrogen molecules to produce atomic hydrogen. The atomic hydrogen combines with oxygen to form hydrogen peroxide. Losses are encountered due to recombination of hydrogen atoms on the walls of the vessel, and to deactivation of mercury by oxygen.

In the experiments performed, dry gaseous hydrogen and oxygen were supplied through calibrated flowmeters, mixed, and the mixture passed over mercury at 100 C. The gases were then cooled to room temperature and passed through a 20-mm ID quartz tube exposed for a length of 25 cm to radiation from a mercury-vapor lamp. The condensable products were collected in a liquid air trap specially designed to eliminate fog formation. The quantum input to the reaction tube was calibrated by using uranyl oxalate as an actinometer, and the data on peroxide yield then combined with the quanta input obtained in this way to give the "quantum yield." Values of quantum yield calculated by this procedure were 0.5 or less. If the reaction between oxygen and atomic hydrogen is

$$H + O_2 \rightarrow HO_2$$

and

$$HO_2 + HO_2 \rightarrow H_2O_2 + O_2$$

or

$$HO_2 + H \rightarrow H_2O_2.$$

the quantum yield should be unity. The lower values obtained are believed to be due to the formation of ozone, which would not only consume quanta, but would destroy peroxide directly.

With the straight reaction tube and the straight lamp the best results were obtained using a ratio of hydrogen to oxygen of 3.2. With this ratio of the two gases both total mols of peroxide produced and peroxide concentration in the condensate increased with increase in total gas flow rate up to the maximum rate employed of 5,000 cc per minute. Under these conditions the product concentration was 74 per cent, and the production was 0.017 gram mol per hour.

As in the case of the glow discharge experiments, the observed power consumption was very high. Based on the fraction of the total ultraviolet light absorbed by the reaction tube, the observed power consumption was estimated to be 14 kwh per lb 100 per cent hydrogen peroxide. Based on the quantum energy of 2,537 A light and a quantum yield of 0.5, the theoretical power consumption is 3.6 kilowatt-hours per pound. It seems probable that with proper design the process would operate with a power consumption less than for the glow discharge method, but perhaps greater than for the electrochemical processes. It has the advantage of not requiring liquid air for the condenser, and of producing peroxide at concentrations as high as 85 to 90 per cent directly. The disadvantages are the mercury contamination and the necessity of cooling and recirculating large quantities of gas.

25.4 CYCLIC REDUCTION AND OXIDATION OF 2-ETHYLANTHRAQUINONE

In the spring of 1945 it was learned that the Germans had developed a process for manufacturing hydrogen peroxide by the cyclic reduction and oxidation of 2-ethylanthraquinone,

[redacted]

and were, in fact, proceeding with the installation of very large plant capacity based on this method. An analogous method using azobenzene had previously been developed in the United States, but had not been adopted commercially.

Because of this Section's interest in methods of manufacturing hydrogen peroxide, NDRC Division 9 agreed to sponsor a preliminary laboratory study of the reactions involved in the German process. The laboratory work[3] demonstrated that the cyclic process gave quantitative yields of hydrogen peroxide, and that the cycle may be repeated many times without significant decrease in yield. An important development of this study was the demonstration that by using tetrahydro-2-ethylanthraquinone it is possible to obtain about twice the yield of hydrogen peroxide per cycle as when 2-ethylanthraquinone is used.

25.5 APPLICATION OF MODERN FLUIDIZED POWDER TECHNIQUES TO THE BARIUM PROCESS

25.5.1 Introduction

Although dilute solutions of hydrogen peroxide have been produced by the barium process for more than a century, the electrolytic processes, introduced some forty years ago, proved to be more economical; in the spring of 1945 only about 13 per cent of United States production was by the barium process.

The barium process has been operated with various modifications, but generally involves first the production of barium oxide by decomposition of the carbonate or nitrate and subsequent oxidation of the oxide to the peroxide in a stream of air (or oxygen) at about 600 C. The barium peroxide is then treated with sulfuric acid to form hydrogen peroxide and permit the removal of barium as the insoluble sulfate. The barium sulfate by-product, though not a high-priced chemical, has sufficient value to make the process competitive for the production of hydrogen peroxide in amounts corresponding to the market for barium sulfate.

There are obvious possibilities of modifying this process by recirculating the barium and so eliminating the economic dependence on the sale of the barium by-product. Sulfuric is the preferred acid for reaction with the barium peroxide, since the resulting hydrogen peroxide is purer and more stable than when nitric acid is used. The nitrate is the preferred salt to use in producing barium oxide, since the product is in the desired porous form, making the oxidation to the peroxide relatively simple. In order to complete the cycle various steps may be employed. The sulfate may be converted to the chloride and this in turn converted to the nitrate by double decomposition with sodium nitrate. Much work has been done on the use of barium carbonate, with carbon dioxide employed to react with barium peroxide to produce hydrogen peroxide. Decomposition of the precipitated sulfate has not proved practical, and decomposition of the carbonate has required special techniques to produce the porous form of barium oxide.

It seems evident that the lack of commercial success of the various cyclic barium processes for hydrogen peroxide is due mainly to the difficulty and expense of handling reacting solids at high temperature levels. If the decomposition of either the nitrate or the carbonate could be simplified, the cost of hydrogen peroxide by the barium process could be reduced substantially. Furthermore, if it could be made cyclical, the process would not depend on the market for precipitated barium sulfate, which now represents 25 to 30 per cent of the value of the two products. For these reasons an attempt has been made to apply the relatively new fluidized powder techniques to the problems of barium nitrate decomposition and barium oxide oxidation at high temperatures, with a cheap cyclic process as the objective.

25.5.2 Application of Fluidized Powder Technique

By the phrase "fluidized powder technique" is meant a continuous method of contacting a gas and a finely divided solid. The mass of powder "floats" in a stream of gas passing vertically, powder concentrations high enough to produce mixture densities of 20 to 50 lb per cu ft, or even greater, being attainable. Because of the turbulence of the stream the churning bed of powder tends to be mixed rapidly, and the gas is brought into intimate contact with the large surface of the solid. Both gas and powder may be fed and withdrawn continuously.

In order to explore the applicability of this technique to the barium peroxide process, experiments were carried out in two laboratory reactors 1⅜ and 2 in. ID respectively, each 4 ft tall. These were made of alloy steel and electrically heated to controlled temperatures. The powder could be fed continuously either at the top or bottom. The hot gas leaving the top passed to a cyclone separator from which solid fines were recovered. In tests of barium nitrate decomposition the nitrogen oxides were absorbed by concentrated sulfuric acid in a train of absorption tubes. The gas velocity in the heated reactor was kept within the range of 1 to 1.5 fps (superficial). The gases used were oxygen or dry air free from carbon dioxide.

The most important purpose of the tests was to determine the behavior of the suspended bed of powder, i.e., the "fluidization" and reaction rate characteristics. Satisfactory fluidization of both oxides and the nitrate was obtained at room temperature. As the temperature was raised to 300 C, however, the oxide particles tended to agglomerate and settle out, allowing the gas to channel up through the bed with very little mixing. This effect was due to the fact that the barium oxide, reported to have a melting point of 1923 C, became slightly sticky at quite a low temperature.

In order to avoid this difficulty an inert carrier was introduced in the form of powdered, fused magnesia or a powdered tertiary barium phosphate. Both of these gave satisfactory fluidization, although the phosphate showed some tendency to cling to the walls and settle out. In a typical experimental test the carrier was introduced first and the barium nitrate then added continuously at the maximum rate compatible with good fluidization characteristics. In this way the capacity for barium nitrate decomposition, at 800 C, was found to be about 7.6 pounds per hour per square foot of cross-sectional area in the 2-in. reactor. Since the wall effect and tendency of the powder to bridge across the tube is much less in large reactors, the capacity may be expected to be greater in reactors of commercial size. The nitrate decomposition reaction was very slow at 400 C but was very rapid and essentially complete at 800 C. Best results were obtained by feeding the nitrate at the bottom of the bed. Finely divided fused magnesium (through 100 mesh) was found to perform well as the carrier, and steady nitrate decomposition was obtained when the nitrate feed was fed at the bottom mixed with from 5 to 20 times its weight of carrier fines from the cyclone separator. The data on absorption of nitrogen oxides in the gas stream leaving the reactor indicated that a negligible fraction of these gases was reduced to nitrogen and that their recovery as nitric acid should be practical by conventional means.

The barium product of the nitrate decomposition at 800 C was primarily oxide mixed with a few per cent peroxide (mixed with magnesia carrier). Conversion of the oxide to the peroxide in air or oxygen takes place rapidly and completely at about 600 C, and this part of the cycle appears to present no serious difficulties if the fluid powder technique is employed with an inert carrier powder.

25.5.3 Formation of Dilute Hydrogen Peroxide from Barium Peroxide

The use of sulfuric acid in this step is well established but is undesirable because of the difficulty of recycling barium sulfate. Using phosphoric acid at 0 C and a *p*H below 4, 95 per cent conversions were obtained at hydrogen peroxide concentrations of 2 to 3 per cent.

Conversion of barium peroxide to hydrogen peroxide was also shown to be practical using nitric acid, thus directly producing barium nitrate which can be reconverted to the peroxide. Conversion of the peroxide so produced was 82 to 87 per cent at *p*H of 1 to 3 at 0 C in hydrogen peroxide concentrations of 2 to 2.5 per cent. Conversion was greater at the lower peroxide strengths. A continuous system for carrying out the reaction was described. Loss of

[redacted]

carrier powder (fused magnesia) due to solution by nitric acid was shown to be unimportant. The separation of the peroxide from the barium phosphate or barium nitrate in solution was not studied carefully, but appears to be practical. Concentration of 2 to 3 per cent solutions up to any desired strength can now be considered as entirely practical, and not expensive.

25.5.4 Conclusions Regarding Barium Peroxide Process

The investigation outlined was inadequate as a basis for appraising the full potentialities of a barium peroxide process utilizing the new fluid catalyst techniques. It did establish, however, that barium nitrate can be decomposed completely using a continuous powder system with an inert carrier powder, evolving the nitrogen in recoverable form, and that the oxidation of barium oxide to peroxide can also be carried out in a continuous powder bed. The development of a cyclic operation appears to present no serious obstacles, and it may be that this can be based on barium nitrate, with recovered nitric acid used to react with the barium peroxide. Further laboratory work may indicate the desirability of using two acids, probably phosphoric for the peroxide reaction and conversion of the phosphate to nitrate prior to barium oxide production.

The basic principles of a practical cyclic process appear to be established. Such a process would require only fuel, water, air (or oxygen), and a small fraction of the power required by the electrolytic processes. The total of fuel and power costs is estimated to be less than 1 cent per pound of concentrated hydrogen peroxide. Based on laboratory rate data, the total power and fuel requirements of the process are estimated to be 0.5 kilowatt-hour and 400,000 Btu, respectively, per pound of 100 per cent hydrogen peroxide.

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Chapter 26

IMPROVEMENT OF SHOES

26.1 SUBSTITUTE SHOE SOLES

THE OBJECT OF THIS investigation was to find satisfactory substitutes for leather used as midsoles, and for GRS used as outsoles in Army shoes. In December 1943, because of the critical shortage of leather, it appeared that some substitute materials must be developed if the Army requirement for shoes was to be obtained.

Promising materials were secured from manufacturers by maintaining a field representative to visit them. Results obtained by tests were reported to the manufacturer with recommendations for improvement. Over 200 samples were obtained and tested.

26.1.1 Laboratory Tests and Approximate Standard Values

Laboratory test methods were studied and a set of standard tests developed which included stitch-tear strength, tensile strength, water absorption, effect of high and low temperatures, abrasion resistance, and flex-pressure resistance.

Thickness. Measurements were made with an Ames dial gauge. Army shoes should have a thickness of 8 iron or more for each sole.

Density. Determined by weighing and displacement of a mercury column. Leather shows about 1.02 grams per cubic centimeter. All other factors being satisfactory, the lower the density, the better; values above 1.3 would be excessively high.

Stitch-Tear Test. Determined according to American Leather Chemists Association [ALCA] standards, using a Scott testing machine. Government specifications require a minimum strength of 30 pounds dry and 25 pounds wet. Since thickness is obviously a factor in this test, the values calculated in pounds per iron should be above 4 pounds dry and 3 pounds wet.

Tensile Strength and Deformation. The samples used were die-cut to ALCA standards, with a necked section ½ in. wide and 4 in. long; these were broken on a Scott machine and a stress-strain curve drawn automatically. A tentative minimum satisfactory value is about 1,200 psi.

Water Absorption. The sample was conditioned for 5 hours at 55 C, cooled, and weighed; immersed in water at about 25 C for 24 hours, drained, and weighed. The difference was water absorbed. The sample was reconditioned for 5 hours at 55 C, cooled, and weighed to obtain water-soluble materials. Satisfactory midsoles and outsoles have a low water absorption. Leather absorbs about 70 per cent, which is not satisfactory, under 10 per cent being more nearly ideal. Water-soluble material lost should be as low as possible, and not over 2 per cent.

Cold Cracking. Samples were conditioned for 24 hours at −20 C and bent 180 degrees over a mandril starting with a 3-in. diameter. The diameter over which the sample cracks is recorded. Any diameter less than 1½ in. should be reasonably satisfactory.

Abrasion Resistance. Determined on a modified Taber abrasion machine. Satisfactory leather outsoles wear about 0.9 gram per 1,000 revolutions, and the present GRS Army outsole about 0.4 gram per 1,000 revolutions.

Flex-Pressure Test. A new type machine was developed simulating the bending and rolling action under pressure undergone by a shoe sole in walking. A material not disqualified by excessive elongation is considered (1) excellent, if it completes 100,000 cycles; (2) fair, if it completes 50,000 cycles. When a material showed exceptional promise, it was given a flex-pressure test under a constant stream of water. Under such conditions, 50,000 cycles was considered excellent.

High-Temperature Tackiness. Samples subjected to 55 C for 5 hours should show no tackiness.

[redacted]

A complete description of these tests and the results obtained on all samples are reported in the Final Report.

26.1.2 Summary of Results

Extensive abrasion tests on GRS outsoles from different manufacturers disclosed wide variations between the products from different factories; the best was more than twice as good as the poorest. It was also found that, in general, the wear resistance of the toe portion (where the greatest wear resistance is desired) was lower than that of the shank and heel portions. These results were discussed with the manufacturers resulting in improvement in the quality of the outsoles being furnished to the Army.

Laboratory examination of the samples tested disclosed two substitute midsoling materials and two substitute outsoles which were outstanding. Both of the midsoles are paper-base products impregnated with and bonded together by plastic compositions.

One of these products, although laminated, showed no signs of delamination under the most strenuous test conditions. It showed water adsorption somewhat greater than leather, which is not to be desired, but withstood flexing under pressure while wet much better than leather. The abrasion resistance was poor, but a midsole is not required to show high abrasion resistance.

The other midsole material was not laminated. Its water absorption was low, about 10 per cent, and its abrasion resistance of the same order as leather.

The outsole products both consisted of a vinyl plastic sheet in which was incorporated a fiber base. In one product the fiber base was a carpet-weave fabric and in the other it was a loose felt. The latter is to be preferred as it gives a smoother edge finish to the shoe sole. Both these outsoles had all the desirable characteristics for good soles and were particularly outstanding in their abrasion resistance, which indicated that they should outwear leather six or eight to one.

Civilian wear tests corroborated the laboratory findings and several hundred Army shoes were built for a test at Camp Lee. As of March 1946 this test still was in progress. The results obtained to date are encouraging.

On the basis of these facts it is recommended that both the midsole and outsole materials described be used in Army shoes.

26.2 ADHESIVES

The objects of this phase of the investigation were: (1) to obtain adhesives which would give a permanent bond between the leather midsole and GRS outsole of Army shoes, and (2) to obtain an adhesive and method for cementing the insole lip of oil-treated insoles.

When GRS outsoles are sewed to leather midsoles, as is done in the present type of construction of Army shoes, there is a tendency during wear for a pocket to develop between the two which allows water and mud to accumulate. It was felt that if these two soles were cemented together the shoe would be much better and show longer life.

Since the majority of Army shoe failures appear to be due to disintegration or malformation of the insoles under wear conditions, a method was developed at the University of Cincinnati for oil treating insoles which would approximately double their life. A manufacturing difficulty arose, however, in the manufacture of these oil-treated insoles into shoes, as it proved to be impossible to form the insole lip with the adhesives generally used for that purpose. It became necessary, therefore, to devise some new method for this manufacturing operation. A new adhesive was needed which would work on oil-treated leather insoles, and it was desirable that it be not critical as to drying time, thereby eliminating all elements of employee judgment as to when the parts were ready to be put through the lip forming operation.

It was obvious, from investigation, that no adhesives presently used in the shoe industry could be relied upon to accomplish either the sole-cementing or the lip-cementing jobs. Other industries, therefore, were investigated, such as the automotive, airplane, and plastics. A number of adhesives entirely foreign to the shoe industry were discovered, and these were tested to evaluate them for the problems at hand.

This involved setting up a standard procedure for evaluation of adhesives in the laboratory, as no accepted standards were available in the industry. This was done, and is fully described in the Final Report. Preparation of the sample, application of the adhesive, and the test methods were all of importance.

Over 70 samples of adhesives were tested in the laboratory, and the results obtained with each are reported. The lip-setting operation involves a leather to leather bond under extremely short pressure dwell, while the midsole to outsole problem involves the bonding of leather to GRS. Both were tested for all samples, and the best adhesive determined.

26.2.1

Summary of Results

The results of laboratory work indicated that the strongest bonds could be secured by applying the adhesive, usually in two coats, allowing it to dry completely, and then heat activating the surfaces just before they are pressed together. By this method it was possible to avoid any critical drying time until the adhesive became "tacky," as is the case with adhesives now in use in the shoe industry.

It was also found that the best adhesives, made by several manufacturers, were all of the same general type, containing Buna N as a principal ingredient. Solvents are important, and some evidence exists that the useful shelf life of these adhesives depends largely upon the use of proper solvent combinations.

Adhesive bonds obtainable with the latex-type adhesives at present in use in the shoe industry have strengths of 10 lb or under per in. of width. With the adhesives of the new type, heat-activated bonds of 30 lb or over per in. of width may always be obtained either for leather to leather or leather to GRS. Results as high as 92 lb per in. of width were obtained in the laboratory tests. The cementing of GRS outsoles to leather midsoles by this method and using the recommended adhesives is entirely possible. In fact, bonds may be obtained which make stitching unnecessary.

Laboratory tests on lip setting resulted in the development of a new method for which patent application has been made. In this method the adhesive is applied over the channelled insole and allowed to dry. The lip is then set in a standard lip-setting machine which has been fitted with a jet, causing hot air to impinge upon the adhesive-coated surfaces of the lip just before they enter the rollers which pinch them together. The speed of the machine is such that only the surfaces of the adhesive are heated to the activation point, the leather itself remaining cold.

Lips formed by this method proved much stronger than those made by the conventional method. In addition, there was no difficulty in using these adhesives and the hot-air jet method for lip setting of oil-treated insoles. In fact, it was found that the adhesive could be applied to the insoles while still wet from the oil treatment and then dried.

A factory test was made at the Army Shoe Rebuilding Plant at Hannibal, Missouri, which was completely satisfactory. Several hundred pairs of insoles were oil treated, the adhesive applied, dried, and subsequently lip-set by the hot-air jet method. All were satisfactory and were built into Army shoes.

As a result of this work, it was recommended that Army shoes be built with the GRS outsoles cemented to the leather midsoles using one of the approved adhesives and heat activated. Also, it was recommended that the hot-air jet method of lip setting be adopted on Army shoes.

Chapter 27

IMPROVEMENT OF LEATHER

This project on the improvement of leather investigated synthetic tanning materials, mold resistance of leathers, special treatments to improve leather, and methods for increasing water resistance of Army shoe upper leathers.

27.1 DEVELOPMENT OF NEW SYNTHETIC TANNING AGENTS

The objectives of this program were (1) to minimize the possibility of a shortage of vegetable tannins and (2) to obtain better leather through the use of synthetic tannins. The physical wear of sole leather was not so satisfactory as desired under severe conditions. German patents applicable to the problem were reviewed, and synthesis and evaluation of 60 examples taken from these patents were accomplished. The influence of the acidity or alkalinity of the system and the weight ratio of the two components upon tanning of the furfural-resorcinol polymers were also evaluated as a basis for future work. The expense involved in the use of these polymers as pure tanning agents was prohibitive. It was shown that, when in combination with waste liquors from the sulfite-cellulose paper industry, a product with very desirable properties was obtained. The various sulfite-cellulose products which are available were studied in order to make possible an intelligent choice of the sulfite-cellulose waste to be used.

Leathers treated with various products were evaluated from a variety of standards, and from this evaluation a group of formulations were selected as worthy of pilot plant study. Conditions were established for the preparation of these formulations in the pilot plant of the Monsanto Chemical Company and arrangements completed for the use of eight of these formulas in the experimental tannery of the American Oak Leather Co.

Although the resistance of glyoxal-resorcinol tannages to alkaline detannization was interesting, the investigation was not carried beyond the laboratory stage.

27.2 MOLD RESISTANCE OF LEATHERS

Whenever leather is used or stored in an atmosphere where the humidity is high and the temperature is 70 F or higher, mold development will occur. All disinfectants adaptable to leather were evaluated and the best method of application determined. The American Leather Chemists Association method was used for evaluation and treatment. From a series of special reports on this subject, it is found that *p*-nitrophenol was a suitable disinfectant. It was approved in limited concentration in dubbin by the Surgeon General. The effect was enhanced when *p*-chloro-*m*-xylenol was used in combination. Shirlan (salicylanilide) was evaluated and found to merit further work. Based on effectiveness, low toxicity, and availability, *p*-chlorophenol and tetrachlorophenol were recommended.

In the use of a mold resistant treatment, care must be exercised in selecting an agent which (1) is effective, (2) is economically and practically available, (3) has no deleterious influence on the leather, and (4) has very low toxicity to persons wearing or handling the leather. No definite knowledge has been collected on the actual damage done by molds and associated micro-organisms. Most of the organisms encountered, except in shoes, were nonpathogens, but the same conditions which permit or cause molds to grow are also conducive to the growth of disease producing organisms.

27.3 SPECIAL TREATMENTS TO IMPROVE LEATHER

The following results were obtained in a study of special treatments to give leather special characteristics or protection. (1) Suitable wax impregnations were developed to give firmness to leather. (2) Synthetic resins available and included in this study improved wear, but were no better than the oil-grease treatment. (3) Tests showed that there is little correlation between resistance to abrasion and actual wear; also resistance to abrasion was found not to be capable of correlation with the chemical analysis of leather. (4) Rapid deterioration of insole leather, characterized by curling, cracking, buckling, and shrinkage, was found to be due to the influence of wetting and drying and the alkaline nature of perspiration. (5) Because of the shortage of brass, aluminum, copper, and other metals, it was necessary to use iron in the Army shoe; iron as such has a definite deteriorating influence on vegetable-tanned leather. (6) Aside from the fact that Canadian tanners had better hides to tan (they made heavier leather) and no limitation on chrome, their leathers were by analysis no better than ours; shoes made of these leathers and tested in actual service at Camp Lee substantiated this conclusion. (7) The influence of perspiration on insole leathers, the influence of alkaline soil solution and its penetration into flesh out leathers, and the prevention of shrinkage of leather midsoles were also studied.

27.4 STUDIES TO INCREASE WATER RESISTANCE OF ARMY SHOE UPPER LEATHER

This problem was concerned with many other factors, such as water absorption, the rate of flow through leather, and drying characteristics, as well as shoe construction.

Numerous special reports were issued including: a preliminary study of the influence of oils when added to finished leather; a report on Canadian leather; a résumé of the characteristics of Army shoe upper leather; a dynamic water penetration test to determine what leather will do in service; area shrinkage of grain and flesh out Army leathers; impregnite (an oil, grease, etc., composition developed by the Chemical Warfare Service) as an equal to any other compound or material evaluated for water resistance; water penetration tests of shoes, in which not much difference was found between shoes of grain out and of flesh out leathers; evaluation of lanolin for water resistance; influence of hardness of greases; influence of resins and other materials.

It is believed that only by a knowledge of the fundamentals of skin chemistry and tanning mechanism can a leather with a maximum water resistance be produced.

Chapter 28

DELEADING OF GASOLINE

28.1 INTRODUCTION

THE PURPOSE OF this investigation was to devise a convenient and rapid method for deleading Army gasoline. Two methods for accomplishing this purpose were developed; they are called, respectively, the "fast" and the "slow" (overnight) processes.

28.1.1 Fast Process

The first step in the fast process is to treat the leaded gasoline with tin tetrachloride. The mixture is then treated with a slurry composed of ligroin in which triethylamine is dissolved and powdered activated charcoal is suspended. The triethylamine combines with the lead and tin compounds to form flocculent insoluble products which are adsorbed on the charcoal. The deleaded gasoline is then separated from the charcoal and adsorbed material by decantation.

By use of the fast method, 5 gallons of gasoline can be almost completely deleaded within 40 minutes. The gasoline recovered in deleaded form is 95 per cent of the leaded gasoline used. If the leaded gasoline contains a low percentage of unsaturated hydrocarbons, the gum content of the gasoline is not much increased. When gasoline containing a high precentage of unsaturated hydrocarbons is used, the increase in gum content is greater, but still not enough to be harmful.

28.1.2 Slow Process

The gasoline is treated with triethylamine perhydrobromide $(C_2H_5)_3NHBr \cdot 2HBr$ in the slow process. A flocculent precipitate is formed. Powdered activated charcoal which adsorbs this precipitate is added. Then the deleaded gasoline is separated from the charcoal and adsorbed material by decantation. The reason for the slowness of the process is that, after the addition of the perhydrobromide, the gasoline must be allowed to stand in contact with the charcoal for at least 10 hours in order to secure satisfactory removal of the lead.

28.2 RESULTS

After the "fast" process had been developed, kits designed for field use were planned in cooperation with the Quartermaster Corps. Results of laboratory experiments with this method, and field tests with the kits have established the following facts:

1. The deleaded gasoline contains:
    - a. An amount of lead equivalent to 0.05 to 0.20 ml of tetraethyl lead per gallon.
    - b. Less than 20 mg of gum per 100 ml of relatively saturated gasolines, but between 100 and 200 mg of gum per 100 ml of highly unsaturated gasolines.
    - c. No compounds of tin.
    - d. No coloring matter. (The deleaded gasoline may thus be easily distinguished from the original material.)
    - e. No substance which corrodes copper or steel.

2. When the deleaded gasoline is withdrawn from a 5-gallon "blitz" can, there is left in the can 1,000 to 1,500 ml of gasoline and charcoal sludge. Thus 90 to 95 per cent of the gasoline is recovered in deleaded form. After the sludge is dumped, the can is ready for use in deleading another lot of gasoline.

3. A liter or two of deleaded gasoline is obtained at the end of 20 to 30 minutes. The entire 5 gallons of deleaded gasoline (minus the 1,000 to 1,500 ml left behind with charcoal sludge) is obtained in 30 to 45 minutes. Within this period, only about 10 minutes of actual labor is required. The remainder of the time is consumed in draining the can, etc.

28.3 CONCLUSIONS

With gasoline containing only small amounts of unsaturated hydrocarbons, the slow process gives results which are equivalent to those obtained by the use of the fast process. When gasoline containing larger amounts of unsaturated hydrocarbons is used, the slow process fails to remove considerable portions of the lead and increases the gum content. For this reason, this process is not recommended for use with gasoline of this type.

Chapter 29

EVALUATION PROCEDURES FOR WATER-REPELLENCY TREATMENTS

29.1 SUMMARY

The importance of water-repellent clothing has been recognized for some time. Since no methods were available for quickly predicting the service that would be obtained from water-repellent clothing under the severe conditions of military use, an investigation of test procedures was necessary. The purpose of the investigation was to evaluate the procedures for measuring water-repellency treatments and to correlate laboratory studies with Army field tests. The scope of the investigation included new test procedures, evaluation of various water-repellent finishes, the durability of water-repellent finishes to wear and laundering, the effect of detergents and sea water on water repellency, and the influence of air permeability, of the nature of the fabric, and of underlayers on resistance to penetration by rain.

29.2 EVALUATION OF TEST PROCEDURES

29.2.1 The Drop-Penetration Test

The apparatus consisted of a drop former, a support for the fabric to be tested, and an electrical signaling device to indicate failure of the fabric. The drops issued at a rate of one per second from each of 31 capillaries and fell 5 ft 8 in. through a tube (to protect them from air currents) before striking the sample. The drops had a volume of 0.075 ml. The sample was held under slight tension over a backing of 12 layers of absorbent gauze. The latter was supported by a transparent plastic plate held at a 45° angle. When water passed through the sample, the gauze was moistened, and an electric circuit was completed between the wires of a grid embedded in the surface of the plastic plate. When the circuit was closed, a buzzer sounded to indicate the end point.

In tests with natural rainfall, samples of the water-repellent fabrics, mounted as in the drop-penetration test, were exposed and the penetration of water was observed by the use of a water-soluble dye in the backing. The results of the drop-penetration test correlated satisfactorily with the results of tests with natural rainfall.

The other methods of evaluating water repellency do not in every case correlate with the results of drop-penetration tests or with natural rainfall. This result was illustrated by tests with water-repellent 5-oz poplin and 13-oz jungle cloth. Even where there existed corresponding variations in the results of other tests, the drop-penetration test was a more sensitive measure of differences between fabrics; e.g., the samples containing 1.2, 2.5, and 3.5 per cent, respectively, of a nondurable finish differed only slightly in their air permeabilities, spray ratings, per cent absorption, and hydrostatic pressure values, whereas they showed rather large differences in their drop-penetration times.

In addition to factors inherent in fabrics, variables inherent in the backing influence the resistance to penetration by falling drops. When the water-repellent samples were backed with a plate of transparent plastic, it was noted that the first drop to strike the fabric invariably penetrated, owing apparently to the almost complete lack of cushioning effect, so that most of the energy of the drop was available for penetrating the fabric. With a sweat shirt backing, the initial penetration was observed in practically the same time as with no backing, while longer times were required when gauze compress or blotting paper was used. Although they contained sufficient viscose so that they absorbed water rapidly, the two serges tested as backings caused a marked increase in the time required for initial penetration. Experiments showed also that, for the absorptive materials used as backings, the time required for initial penetration was at least in part related to the contact made between the water-repellent fabric and the backing.

Moisture in the backing caused an important reduction in the resistance to rainfall of a water-repellent fabric, but the water repellency of the thoroughly wetted fabric was not restored when it was tested over a dry backing.

The observations on the influence of the backing on the results of drop-penetration tests suggested the use of two layers of water-repellent fabric.

In cases where increased severity of the test was required, a modified drop-penetration test (gauze backing omitted) or the du Pont Rain Test was used. In this test the fabric is subjected to heavy streams of water for 5 minutes at heads of 2, 4, 6, and 8 ft, the amount of penetration being measured at each head, and the fabric is rated according to the sum of the weights of water passing through at these four heads.

29.2.2

Other Test Methods

Surface tests included spray rating, "pearl" tests, and contact angle measurements. Absorption tests were useful in indicating how much water was taken up by a finished product. There was no direct relation of these tests to the protection against rainfall. Water-permeability tests were especially useful for testing fabrics designed for immersion suits, water buckets, etc. Of all the conventional tests, hydrostatic pressure tests appeared to correlate most closely with rainfall, particularly when a series of fabrics of the same weight and construction were compared. A high hydrostatic pressure value almost invariably meant good resistance to rainfall, but the converse was not always true.

29.3

DURABILITY OF WATER-REPELLENT FINISHES

Poplin was treated with nine durable type finishes and subjected to wear under comparable conditions, i.e., all factors pertaining to fabric and garment construction were made as constant as possible. From tests on these treated pieces, it was concluded that the relative durability of finishes applied to cotton goods, which will be subjected during their service life to wear and laundering only, can be quickly determined by tests before and after a number of launderings.

29.4

REDUCTION OF WATER REPELLENCY RESULTING FROM LAUNDERING

Experiments were performed to determine the effect of laundering with various detergents: a high titer soap, Nacconol NRD, Igepon T, du Pont MP-189, and Neutronyx No. 33. In the reports which cover this work several tables of data are presented, and the results indicated that the small amount of detergent remaining in a fabric after laundering should not seriously affect its water repellency.

29.5

REDUCTION OF WATER REPELLENCY RESULTING FROM IMMERSION IN SEA WATER

Samples of poplin treated with nine durable water-repellent finishes were agitated in the surf of the Atlantic Ocean for 1 hour, at the end of which the samples were thoroughly wetted. The fabrics were allowed to dry on the sand without wringing, and then were subjected to the spray, hydrostatic pressure, drop-penetration, and air-permeability tests. They were retested after two 10-minute rinses in conditioned fresh water at 100 F. The drop-penetration times of all unrinsed samples were seriously reduced as compared with the untreated controls. The rinsed samples were all higher in air permeability than the respective unrinsed materials, suggesting that the salts deposited in the unrinsed fabrics were in part responsible for their reduced air permeabilities. The results suggest that, for at least three of the finishes examined, the repellency of fabrics immersed in sea water can for the most part be restored by rinsing with fresh water.

29.6

FACTORS THAT AFFECT THE VALUE OF WATER-REPELLENT FABRICS

The value of water-repellent fabrics depended in large measure on fabric construction as well as the nature of the finish. The water-

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permeability test, like the hydrostatic pressure test, measured a quantity that is affected by both the fabric construction and the finish. The air permeability test measured only the tightness of weave of the fabric, and is not affected by the finish. Differences introduced by variation in twist factor are overshadowed by small differences in construction and possibly in finish.

29.7 THE USE OF TWO LAYERS OF WATER-REPELLENT FABRICS

An important improvement in the protection afforded by water-repellent garments can be obtained by the use of double-layer construction. The resistance of penetration shown by two layers of water-repellent fabric was many times as great as that of a single layer. A double-layered garment consisting of a tightly woven water-repellent outer layer and a thick, resilient, water-repellent inner layer would represent a very desirable construction. On the other hand, the use of absorbent linings in water-repellent garments was contra-indicated.

29.8 CONCLUSIONS

The results of this investigation indicated the following conclusions.

1. The relative protection afforded in actual rainfall by a series of water-repellent fabrics can be estimated by means of the drop-penetration test.
2. For a series of fabrics of similar weight and construction, the results of the hydrostatic pressure test correlate closely with results of the drop-penetration test.
3. The relative durability of water-repellent finishes to field wear can be estimated by measuring their repellencies before and after suitable laundering procedures.
4. The deterioration of water-repellent finishes as a result of laundering is caused in large measure by the mechanical action they sustain in the wet state, rather than from specific effects of the detergent used.
5. Resistance to penetration by rainfall is affected to a marked degree by fabric construction, especially in the range of the tighter fabrics now used for military purposes. Even a slight improvement in tightness of weave may result in a very large improvement in resistance to rainfall.
6. The use of double-layer constructions, especially with a resilient, water-repellent under layer, results in a very large improvement in the efficiency of water-repellent garments.

Chapter 30

INSECTS AND OTHER ANIMALS OF INTEREST

30.1 SUMMARY

The final report is an exhaustive compendium of the insects reported to damage articles of the character represented by Quartermaster items. The data are derived from the consideration of material in thousands of papers, examined by the author either in the original or in abstract. This report is intended to serve as a basis for the selection, by the proper authorities, of a series of control measures and for the establishment of a control organization, which should lead as far as possible to the elimination of damage from insect attack.

30.2 INTRODUCTION

It was necessary to consider the substances of which military equipment and supplies are composed and to consider the various methods used in packing and storing equipment. In general, everything of animal and plant origin (wood, fiber, fur, adhesive, or food) was considered in the form in which it was likely to be used in military operations. Also included is a considerable number of insects found in buildings, although no definite information on their food is available.

30.2.1 Kinds of Damage Done by Insects

Practically all the insects considered do their damage by actually biting off and chewing the material; hence, whether or not they digest any of the material, they still effectively remove it. A few insects, notably roaches, spoil food by merely passing over it, since they leave behind substances of disagreeable odor. The main classes of organisms which do appreciable damage are mammals, the insects and arachnids, and the fungi.

30.2.2 Relations of the Animals to the Material Damaged

Insects may be classified as permanent pests (material infested as long as it has adequate food or shelter value), bark pests (beetles infest wood continuously as long as it is covered by bark), one-generation pests (material must be in some special state of curing in order to be infested; insects go through life cycle and then seek new material), secondary pests (these follow damage by primary insects or are attracted by some microorganism which is the source of primary damage), temporary pests (carpenter bees and wasps), accidental pests (attack inedible material or edible material out of the ordinary range of an insect's food, e.g., metal), and predators and parasites (do not actually damage the material on their own account).

Basic biological information on insects covers their structure, food habits and food getting, reproduction and development, requirements for existence, distribution, and dissemination.

30.3 MATERIALS ATTACKED AND NOTES ON THE DIAGNOSIS OF DAMAGE

Under each of the 52 groups of materials those organisms are indicated which are most frequently found as pests and, where possible, the type of damage for which they are responsible; the distribution and expected intensity of damage; the significance of infestations of the particular group of materials in relation to infestation of other groups of materials. Also shown is the relation of the infestation to the use of the infested material, where use can be made of it to give clues to personnel in the field to determine causes of damage. Included in this discussion are foods and closely related materials, equipment and supplies, and their raw materials, materials of construction, metals, houses and storehouses.

30.3.1 Systematic Discussion of the Animals

The organisms considered are arranged in what a biologist considers systematic order. This discussion covers the following insects: Thysanura, Orthoptera, Dermaptera, Isoptera, Embioptera, Psocoptera, Ephemeroptera, Trichoptera, Lepidoptera, Coleoptera, Hymanoptera, and Diptera; Mites, Teredos, and Mammals (rodents and carnivores).

30.4 SUMMARY OF CONTROL METHODS AND SUBSTANCES

Insect infestation may be prevented by separating the insects from the material to be protected by an impassable barrier (e.g., by using a copper shield between concrete foundation and the wood of a building or by using insect-tight glass or metal containers for food); poisoning the material (impregnation of wood with creosote); substituting immune materials (e.g., concrete instead of wooden posts under buildings); and environmental control (cold storage of furs). After insects have reached and started to multiply in the material, they may be killed by physical methods (heat, preferably at least 140 F); poison (baits, sprays, solid contact poisons, and fumigation); and biological methods, utilizing certain predacious or parasitic organisms which attack the species of insect. A brief review is presented of some of the methods recommended for each group of animals.

30.4.1 Geographical Summary

A brief description is given of each of the following natural geographic regions into which biologists have divided the world, and in which it is supposed that a large group of Army personnel will eventually be operating: North America (The Northeast, Southeast, West Coast, and Alaska), Central America, West Indies, South America (Northern Coastal Region), Europe and North Africa, Africa (Eritrea, West Africa), Asia (Western Asia, India, Burma and Malaya, Southeastern and Eastern Asia), Dutch East Indies (Dutch Islands, west of Wallace's line, The Philippines, islands from Wallace's line to western New Guinea), Australasia (New Guinea, Tropical Australia, and Temperate Australia), the Pacific Islands (Micronesia, islands east of New Guinea, New Caledonia, New Zealand, etc., the Fijis, Central Pacific Islands, Hawaii, and the Galapagos).

30.4.2 Conclusions

The Appendix to the Final Report lists approximately 1,050 species of insects and mites which either are known to damage materials of interest to the Quartermaster Corps, or very similar substances, or have been found in buildings and are related to destructive insects. It is noted that the most important natural groups of insects are beetles, termites, and moths; however, in the case of fresh meat the flies play the major role and in the case of buildings ants are most dangerous.

In cases where infestation begins before food was packaged, the incidence of such damage can be diminished by careful selection of stock, cleanliness, control to prevent building up of insect population, and final heating of package after sealing to kill any insects which may still be present. As yet no universal package has been developed which will be proof against invasion by all insects.

Equipment and supplies can be protected, while in storage, by using closed containers, *p*-dichlorobenzene, cold, and buildings immune and unattractive to insects. When the equipment is being used, its protection will have to depend on insectproofing or the use of materials which are themselves inedible; unfortunately, relatively few of the possible materials such as synthetic fibers or plastic substitutes for leather have been tested to determine the effect of insects upon them.

Buildings and materials used can be protected against insect attack by structural, chemical, or environmental means. The protective effect of metals resides almost entirely in the physical character of the particular metal, i.e., if the metal is soft enough it will be penetrated by insects.

The criteria for choice of a poison involves not only the efficacy of the poison in question in connection with a given method of application to the pest, but also the disturbance of use of the building which is involved, and the toxicity to man.

Practically all insects and arachnids can be killed by heating at 160 F for 20 minutes; storage of materials at a temperature under 50 F will normally prevent any increase in the number of insects present. According to the few references available relating to the minimum moisture required in food to be packaged, the range is usually 6 to 10 per cent. Mammals must be attacked by specially prepared and placed baits, or with traps in case of rodents.

30.4.3 Recommendations

The following recommendations are made. (1) Obtain and disseminate information on which control measures may be based. (2) Issue regulations and manuals covering at least the basic items in the supervision and performance of pest control. (3) Develop means of obtaining the most effective actual operations against insects and rodents. (4) Write specifications aimed toward obtaining certain raw and processed foods, as nearly as possible free of infestation when delivered to the Quartermaster Corps. (5) Develop means of testing, both in the laboratory and in the field, the efficiency of such substances and methods as may be proposed for pest control, as well as determine the possibilities of insect attack on newly proposed or invented materials, such as synthetics. As a corollary, there are included substances applied as preservatives against other conditions, such as mildew and rot.

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Chapter 31

TROOP FEEDING PROGRAMS

31.1 SUMMARY

A COMPREHENSIVE INVESTIGATION of troop feeding in the United States Army from the Revolutionary War (1775) to the beginning of World War II (1940) is presented. The project, undertaken to provide historical perspective for the evaluation of current subsistence problems, is discussed in the following sections: I. Establishment of the Ration for Troops and Legislation Pertaining Thereto, 1775-1789; II. Legislative Changes Affecting the Ration, 1789-1818; III. Subsistence and Rationing under the Commissariat, 1818-1861; Supplement: Army Regulations and Revised Statutes; IV. The Ration in the Period of 1861-1912, including the Civil and Spanish Wars; Supplement: Army Regulations and Special Statutes; Appendix: Quotations on Use of Ration by the Soldier; V. Subsistence and Rationing under the Quartermaster Corps; Appendix: The Work of the Food Division, Sanitary Corps.

31.2 HISTORY OF TROOP FEEDING IN THE AMERICAN ARMY

A general review of the methods of providing subsistence for troops of the American armies either in active warfare or in periods of inactivity is combined with a résumé of the more important legislation necessary to authorize and fix procedures and standardize rations.

Since the food supply of the Army and Navy must be drawn from, and be closely associated with, the national food supply and its production, and must be transported, it is apposite to call attention to the dependence on agriculture and facilities for transportation. Our enormous expansion in broad agriculture and animal husbandry has supplied the basic foods needed for the diet of the fighting men. This diet has been greatly broadened in later decades through the specialized developments in food preservation and conservation which have come into practical use during the past hundred years and which have all contributed greatly to the solution of the problems of Army subsistence. Changes in modes of transportation from the oxcarts of the Revolutionary days through the period of mule- and horse-drawn carts to railroad and motor truck have done much to solve distribution problems. The development of canning as applied to meats, vegetables, fruit, and condensed or evaporated milk; the wholesale partial drying of fruits and vegetables; refrigeration of meats and other products; and dehydration of milk, eggs, meats, etc., have been of vast significance in improving the available diet for men in service and in making possible the economical transportation of foods. Thus it can be seen that the matter of troop feeding in modern warfare is closely associated with these great commercial and industrial developments.

The general survey of the history of the ration and of troop feeding is presented as a continuous account of the procedures which have been followed in the American Army from its early days. The developments and evolutionary changes which have been made are recorded. Such an account includes a record of early usages, the difficulties encountered in methods of procuring food and its administration and distribution, and the legislation necessary to authorize and accomplish changes which have been found desirable or necessary.

Despite tremendous fluctuations in the demand for food for fighting armies, there has been a certain similarity in the character of the basic foods during the entire period, the staples of bread and meat in some form constituting the two principal ingredients of the army ration. The last quarter century has seen modifications providing a better balanced, more pleasing, and much broader dietary, partly as a result of experience, but to a greater extent because of the improved knowledge of nutrition and of the needs of the body

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under various types of stress. This scientific progress has made it possible and practicable to fortify and improve the basic components of the ration, but has not changed their amounts to an extensive degree, except to make them more compact.

The many changes in the size and character of the Army during the past one hundred and fifty years have made the problems of supply extremely variable, ranging from the feeding of a few hundred men in posts or garrisons in the early nineteenth century to the present tremendous demands for food for millions of men, not only in continental United States, but in various other widely scattered parts of the world, many of which are remote and without normal transportation services. The difficulties in maintaining a regular standardized and consistent method of troop feeding have therefore been varied and often acute. As food must take first place as a munition of war, this situation, enormous in its magnitude, has been met in a remarkable manner.

This review covers not only the nature and quantity of the rations supplied, but also the part which the soldier has had to play in receiving and dealing with them.

A selected bibliography containing approximately 175 references chronologically arranged is given.

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Chapter 32

WESTERN HEMISPHERE BAMBOO AS A SUBSTITUTE FOR ORIENTAL BAMBOO

32.1 SUMMARY

IN ORDER TO LOCATE, in the Western Hemisphere, suitable species and supplies of bamboo meeting the requirements for military ski poles, to be used as a necessary alternative for oriental bamboo now being used, the supply of which has been cut off, a project was originated in the Office of the Quartermaster General. The fabrication of experimental ski pole shafts from various bamboo samples (collected from southern United States, Mexico, Honduras, Canal Zone, Republic of Panama, Colombia, Venezuela, Brazil, Puerto Rico) was undertaken by several manufacturers.

In spite of the fact that it was excelled in strength by several of the other species tested, Tonkin cane remains the ideal material for ski pole shafts.

32.2 FABRICATION OF BAMBOO SKI POLE SHAFTS

The principal steps in the making of documented bamboo ski pole shafts for performance tests are as follows: selecting the material, documenting and marking, shipping, seasoning, sawing culms to length, flaming the culms, sanding the nodes, preparing labels for the transfer of the identification symbol from the culm to the prepared strips, stripping the culms, milling the strips, assembling the strips, mismatching the nodes, cementing the strips with a specially prepared animal glue or a Bakelite compound, wrapping and straightening the shaft, curing the cement, impregnating (some of the shafts cemented with Bakelite compound were also impregnated with another Bakelite compound), and preparing the shafts for performance tests.

32.3 PERFORMANCE TESTS

These experimental ski pole shafts were subjected to the following tests:

The "deflection" test is described in OQMG specification No. 60, Poles, Ski, except that the weight required to deflect the shaft 1 in. in from straight when applied in the center of a 36-in. span, was calculated from a pilot reading based on the actual deflection caused by a 10-lb weight applied in the specified manner. This test was revised in order to prevent possible permanent deflection, caused by the full weight required, which would interfere with subsequent tests.

The "column load" test was conducted in accordance with OQMG specification No. 6C, except that the maximum compressive load sustained by the shaft acting as a column was determined. The test was performed on a Southwark hydraulic universal testing machine of 60,000-lb capacity.

The transverse span-breaking test was conducted according to the specification No. 6C, except that the 8-in. span began 5 in. from the small end of the shaft. This test was performed on the hand-driven screw type Amsler testing machine with pendulum weighing system.

The cantilever test was conducted in accordance with the specification, except that the small end of the shaft was gripped for a distance of 5 in., and the maximum load was recorded at the breaking point.

32.4 CONCLUSIONS

While the number of shafts tested is too small to serve as a basis for final conclusions on many points, the following statements are the result of a conscientious effort to draw from the total experience represented by this project those trends of evidence which seem to be most plain and most significant. Although we have not yet found in the Western Hemisphere a bamboo equal to Tonkin cane (*Arundinaria amabilis,* commercial Chinese origin, taken as the standard of comparison) in all respects, we do have available, in quantities sufficient to meet the requirements envisaged at the time when the project was established, bamboos suitable for the manufacture of ski pole shafts, well above the Army standard minimum performance requirements.

Bambusa tulda excelled Tonkin cane in average performance in all prescribed tests. *Bambusa tuldoides* and *B. longispiculata* excelled Tonkin in average performance in two of the prescribed tests and were both well above the prescribed minima in the other two tests. They are not, however, equivalent to Tonkin in workability or in the type of fracture developed on breaking.

Should the available stocks of Tonkin cane prove inadequate, it was recommended that a portion of the limited supply of *B. tulda* and *B. longispiculata* available in Puerto Rico be used to make shafts during the interval while the machinery was assembled for the exploitation of other species, material for which would have to be brought from more distant places. Although the culms of *B. tuldoides* are somewhat difficult to process because of curvatures which occur naturally, this species, as it occurs in all the States of Rio and Sao Paulo, Brazil, probably is, all factors considered, the best among the species tested for large-scale exploitation.

The three species native to the Western Hemisphere (*Guadua amplexifolia, G. angustifolia,* and *G. superba*) which were tested gave average performances which were above minimum requirements in all but Test 3. *G. superba,* with the best average performance of the three, is also outstanding in its workability and in its low content of superfluous wood. It cannot, however, be recommended for exploitation until opportunity is afforded to investigate its availability and its accessibility in the areas where it occurs naturally. This species is not known to be cultivated on a commercial scale.

G. angustifolia, as it occurs in the valley of the Cauca River, Colombia, is a possible candidate for exploitation, if the minimum requirements under Test 3 are lowered slightly. It is advisable to discard the lower 6 ft or so of culms of these species when selecting material for ski pole shafts, because of the extreme shortness of the internodes in this portion.

Of the species cultivated in the United States, both *Phyllostachys nigra var. henonis* and *Ph. sulphurea var. viridis* showed average performances well above the minimum requirements. Unfortunately, *Ph. bambusoides,* the only one of the three species of the genus tested that is available in any quantity, fell slightly below the minimum requirements for average performance. Also, unimpregnated shafts made from the latter species "take a set" regularly when subjected to moderate but firm lateral stress. In general, the workability of these species was considered to approach most nearly that of Tonkin cane of any of the bamboos tested under this project.

A more complete understanding of the technical peculiarities of the bamboos tested should make possible improved techniques with resulting improvements in performance records. For instance, allowing a longer period for the curing of the culms under natural atmospheric conditions or a controlled artificial equivalent, before initiating the more severe kiln drying, should reduce the incidence of tensions and fractures such as are developed as an apparent result of the prematurely accelerated drying treatment. At least in some instances, the flame treatment of the culms (based on a treatment accepted as standard for Tonkin cane in commercial practice) resulted in definite damage to, and weakening of, the tissues of several of the lots of shafts tested. It was recommended that heat treatments for Western Hemisphere bamboos should not exceed 250 F. It is probable that further experimentation would reveal an optimum range for the heat treatment, or flaming, of the culms for each species.

It is believed to be possible, by giving special attention to technical peculiarities and requirements, to make from selected material of any one of the bamboos tested, with the possible exception of *Arundinaria longifolia* and *Guadua amplexifolia,* ski pole shafts whose perform-

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ance would equal, or nearly equal, the minimum requirements of OQMG tentative specification No. 6C.

The greatest number of failures to meet these requirements occurred under Test 3. This requirement probably could be lowered slightly without revealing any serious deficiency in the quality and "performance in service" of the ski pole shafts.

Although Test 4, as defined in the specification, may have been unsatisfactory, it is believed that the modified execution of this test, as conducted in connection with this project, proved to be a limitation, rather than an advantage. An additional variable was introduced (each of the relatively small lots of shafts had to be divided in order that one-half could be broken in one of the two ways specified in Tests 3 and 4, respectively). Also the important test for "taking a set" was eliminated. With one or two exceptions, the expected correlation between age of culm and performance of shafts made therefrom was not demonstrated by the results of the tests. This may be due partly to the relatively small number of specimens tested and partly to uncontrolled variables.

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Chapter 33

SOLID FUEL FOR HEATING COMBAT RATIONS

33.1 SUMMARY

A SURVEY IS GIVEN OF materials investigated for use in a solid fuel for heating combat rations. Seven types of fuel units were specifically studied, from which a unit composed of trioxane as the fuel ingredient, magnesium stearate as the supporting agent, and carbon black as the pigment, was recommended as the most satisfactory. An alternate fuel of Carbowax 4000 was described. The physical properties, burning characteristics, and methods of formulating and testing were summarized. Recommendations for the manufacture of the submitted fuels were given. A change in the size of the trioxane unit was recommended as a result of field use of solid fuels. Data on the evolution of carbon monoxide from the burning fuels were presented to disprove the initial findings of the Army toxicity tests.

33.2 PURPOSE OF INVESTIGATION

The purpose of this investigation was a study of compositions suitable for the manufacture of individual fuel units for heating of combat rations. The characteristics which such a fuel should possess were given as follows:

1. High heat of combustion.
2. A combustion rate favorable for efficient heating of from 16 to 20 oz of water from 33 to 133 F in from 8 to 10 minutes.
3. Freedom from any volatile toxic product of combustion when the fuel is burned freely, or with reduced air supply, or with the flame in contact with a cold metal surface, or when extinguished by smothering or blowing.
4. Freedom from high initial toxicity; i.e., if the fuel should be eaten by mistake.
5. Free flame of minimum luminosity.
6. Easy ignition at normal or low temperatures by means of one match.
7. Clean burning; i.e., without material deposition of soot or gummy deposits on utensils.
8. Good resistance to wind.
9. Freedom from hygroscopicity and preferably insolubility in water.
10. Freedom from objectionable odors which would contaminate food packed in the same container, and freedom from objectionable odors during burning.
11. A high melting point preferably about 140 F.

It was further stated that the fuel should be a compact, preferably rectangular, tablet which can be packed directly with the ration package.

33.3 RECOMMENDED FUELS

Two fuels were recommended for trial, namely, trioxane and Carbowax 4000. The first of these was represented as most closely fulfilling the requirements while the second was submitted as an alternate made from a commercially available material which approached, but did not fulfill, all specifications. Following is a discussion of the manufacture of each of these two fuel units.

33.3.1 Trioxane, Composition, Weight and Size of Fuel Unit

The following summary gives the preferred fuel compositions. Only formula No. 1 has been used so far to prepare samples and is slightly preferred over No. 2. However, No. 2 is somewhat less expensive because of the lower price and lesser amount required of calcium stearate. Variations of 1 per cent are allowed in these formulations.

	Per Cent By Weight	
	No. 1	No. 2
Trioxane	97	98
Magnesium stearate	2.8	
Calcium stearate		1.8
Carbon black	0.2	0.2

A 30-gram (0.066-lb) fuel unit has sufficient heating capacity (457 btu) to raise the temperature of 1 pint of water 100 F.

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33.3.2 Carbowax 4000

The per cent by weight composition of the Carbowax 4000 fuel unit was calculated to be

89.8 Carbowax 4000
3.2 Ethyl silicate
7.0 Igniter D

However, the composition is better expressed as follows:

100 parts by weight Carbowax 4000
5 parts by weight Ethyl silicate solution S-1053
2 parts by weight of 2.8% ammonia
10 parts by weight igniter mixture

Two of these components are mixtures which are prepared as follows:

Ethyl Silicate Solution. The S-1053 solution is completely hydrolyzed ethyl silicate which is stable for only short periods of time and so is made from a partially hydrolyzed formula (S-1003) which is stable during storage.

33.4 CONCLUSIONS

1. A unit composed of trioxane as the fuel ingredient, magnesium stearate as the supporting agent, and carbon black as the pigment was recommended to the Quartermaster Corps. This trioxane fuel more closely fulfills the requirements for an army combat fuel than any of the others investigated.

2. Sources of the trioxane fuel ingredients, methods of manufacture, burning characteristics, and physical properties were outlined.

3. An alternate fuel of Carbowax 4000 and ethyl silicate was submitted to the Quartermaster Corps.

4. As a result of the field trials on fuels, the following changes were recommended for the trioxane fuel unit.
   a. In order to prolong the burning period, the size of the unit should be altered to provide a thicker cake, changing from $2\frac{3}{16} \times 1\frac{3}{16} \times \frac{1}{2}$ in. to $1\frac{3}{4} \times 1 \times \frac{3}{4}$ in. or $1\frac{1}{3} \times 1 \times 1$ in.
   b. If possible the metal foil wrapper of the trioxane unit should be so fabricated that it can remain around the sides of the fuel during its combustion. This will provide some wind protection and a longer burning period.

5. No satisfactory method was devised for overcoming the faults of the present commercially available fuels: soot and flame luminosity from paraffin wax, necessity of packing alcohol gel in a can, and the toxic fumes from hexamethylene tetramine.

6. If the failings of any one of the commercial fuels are relegated to a position of unimportance, that fuel will become satisfactory for use.

Chapter 34

LITERATURE SEARCH ON CARBONACEOUS FUELS FOR HEATING COMBAT RATIONS

34.1 SUMMARY

A SEARCH OF THE TECHNICAL literature (1920-1943) on the combustion of carbonaceous fuels revealed that charcoal was the most promising for use in a fuel unit to heat army combat rations. The most active catalysts for carbonaceous fuels, i.e., alkali carbonates, would add greatly to the luminosity of the flame, and there were no indications that they would lower the ignition temperature or accelerate the rate of combustion of charcoal to the degree desired in an army fuel unit. Recommendation was made that a highly porous charcoal fuel unit, preferably made of powdered or granular charcoal with a combustible resinous binder, be considered.

34.2 PURPOSE AND SCOPE OF THE LITERATURE SURVEY

This search was undertaken to review published information on the combustion of carbonaceous materials which might be applicable to a fuel unit for heating army combat rations. Special attention was given to catalysts which might provide the essential features of lowering the ignition temperature and accelerating the combustion of these materials. *Chemical Abstracts* were reviewed from 1920-1943 under the following subjects: briquets, carbon, catalysis, catalysts, charcoal, coal, coke, combustibles, combustibility, combustion, fuels, igniters, ignition, inflammability, oxidation, and promoters. Many of the original articles dealing with extensive investigations were read. In addition to information on the most common carbonaceous materials such as carbon, charcoal, coal, and coke, references were collected on certain subjects of interest to the fuel unit problem, i.e., solidified alcohol and metaldehyde.

34.3 DISCUSSION OF THE LITERATURE SURVEY

A discussion of the data compiled from the 536 literature and patent references covered in detail the following subjects: combustion, oxidation reactions, ignition temperature, reactivity of fuels, combustibility, effect of diffusion of oxygen on the rate of combustion of solid fuels, effect of catalysts on ignition temperature, reactivity and rate of oxidation or combustion, mechanism of catalysis in combustion, miscellaneous non-catalyzed solid fuels, briquets (non-catalyzed), and special fuels such as alcohol, polymeric organic fuels, liquid and solidified hydrocarbon fuels potentially applicable to the army fuel unit.

34.4 RESULTS OF THE SURVEY

Combustion and oxidation of carbonaceous fuels:

1. Low-temperature charcoal is the most promising carbonaceous fuel. It has the lowest ignition temperature, highest reactivity, lowest air supply required to maintain combustion, and the most extensive surface.
2. The rate of combustion of solid fuels is limited by the rate of diffusion of oxygen through the stationary gas film on the surface.
3. The most important factors in obtaining low ignition temperatures, high reaction to CO_2, and rapid combustion are low carbonization temperatures and large surface per unit volume.
4. The terms and tests usually employed to evaluate carbonaceous fuels have been designed to indicate their performance in beds of furnaces; no tests indicate their behavior when a match flame is applied to an individual piece of fuel. For heating army combat rations, an individual piece of fuel is required.

5. The results of investigations of ignition temperatures, reactivity, and combustibility of fuels, as well as the effects of catalysts on these characteristics, are not all comparable, because they depend upon the techniques employed by the various investigators.

Catalysis in combustion and oxidation of carbonaceous materials:

6. Alkali carbonates are the best general catalysts for lowering ignition temperatures, increasing reactivity to CO_2, and promoting combustion of fuels in beds. Calcium oxide and iron oxide are less effective, especially in reducing ignition temperatures. In addition, they would add greatly to the luminosity of the flame.

7. The mechanism of catalysis has been indicated to be reduction to metallic sodium or iron in the lower sections of the fuel bed, then vaporization or transfer to the upper bed where the oxide is again formed. The applicability of such a mechanism to small fuel units is not clear.

8. The effect of catalysts on burning rate decreases with the more reactive fuels, i.e., it is less for low-temperature cokes and charcoals.

9. Impregnation is preferable to surface application of catalysts, to avoid shielding and blocking the reaction of oxygen and carbon on the surface.

10. No evidence was found to indicate that any organic catalysts would lower the ignition point and accelerate combustion to the degree desired in the army fuel unit.

34.5 RECOMMENDATIONS

The most active solid fuel available in quantity should be used, i.e., charcoal. Since the limiting factor in combustion is the rate of diffusion of oxygen to the surface, a highly porous structure should be obtained. This might be achieved by providing a number of channels through a charcoal block, i.e., analogous to the form in which smokeless powder is prepared. Alternately, powdered or granulated charcoal might be bonded with a combustible resinous binder. The preferred process would be to form a rigid block without destroying the porosity or blocking the surface of the charcoal. The bonding agent might also be of help in igniting the block. The development of such a fuel should be undertaken by an organization experienced in the manufacture of charcoals and, if possible, of resinous products.

An extensive bibliography was compiled on combustion and oxidation of carbonaceous materials, catalysts in combustion and oxidation of carbonaceous materials, non-catalyzed commercial products, reviews and general information, briquets (non-catalyzed), and special fuels (alcohol, polymeric organic fuels, etc.). There are 536 classified references given.

Chapter 35

FLAMEPROOFING OF TEXTILES FOR ARMY CLOTHING

35.1 EVALUATION OF COMMON WATER-SOLUBLE FIRE RETARDANTS

THE PURPOSE of this investigation was to evaluate the relative flameproofness of fabrics impregnated with common water-soluble fire retardants, using a modified 45 degree microburner flame test and the standard vertical-bunsen flame test.

On the basis of the experimental data obtained, it appears that the modified 45 degree microburner flame test is the superior of the two, since it permits a separation of the better retardants into several groups, the members of each possessing similar flameproofing efficiencies. The standard vertical-bunsen test, on the other hand, is only capable of classification into good, fair, and poor fire retardants.

Considering the results obtained with the 45 degree microburner flame test, the classification of the better flameproofing agents was found to be of the following order of effectiveness:

1. The mixtures of borax-boric acid in the ratios of 7:3 and 1:1, borax-boric acid-diammonium phosphate mixtures in the ratios 7:3:5 and 5:5:1, and the mixture of borax-boric acid-ammonium dihydrogen phosphate in the ratio 7:3:1.
2. Borax-boric acid-diammonium phosphate mixtures in the ratios of 1:1:1 and 1:1:2 and the borax-boric acid-ammonium dihydrogen phosphate mixture in the ratio 7:3:5.
3. Mono- and diammonium phosphates alone and the sulfamate-type fire retardants.
4. Ammonium sulfamate, ammonium molybdate, ammonium sulfate and sodium tungstate.

The above classification is based upon the charred area measurements which represent a measure of the resistance to flaming. The effectiveness in the prevention of afterglow produces a different classification.

The 45 degree microburner flame test is superior to the vertical-bunsen test in that it allows a better separation of the water-soluble flame retardants into groups, the members of each being nearly equally effective. This test has the added advantage of being better able to measure afterflaming of short duration (1 to 3 sec).

Of the several retardants tested, borax-boric acid (1:1) is the most efficient on the basis of the resistance to charring, mono- or diammonium phosphate, the best with respect to afterglow, and borax-boric acid-diammonium phosphate in a 1:1:2 ratio, the best when charred area, flaming and afterglow are considered equally important.

For a given borax-boric acid ratio, the addition of mono- or diammonium phosphate to the mixture decreases the afterglow but increases the charred area slightly.

Mono-ammonium phosphate can be substituted for diammonium phosphate in the borax-boric acid-phosphate mixtures with only a slight decrease in the flameproofing efficiency.

35.2 INVESTIGATION OF THE FLAMEPROOFING OF COTTON FABRICS

The test methods used for the determination of the effectiveness of flameproofing agents and treatments, which have been completely developed, are included in this report. The methods described are as follows:

1. Impregnation of cotton fabric with water-soluble flame retardants.
2. Vertical-bunsen flame test.
3. 45 degree microburner flame test.
4. Laundering test.
5. Leaching test.
6. Tensile strength test.
7. Pyrolysis test.
8. Combustion test.
9. 30 degree flame-rate test.
10. Heating curve measurement (pure retardants).

Experimental methods and equipment for the tests are included. These tests constituted the official methods adopted by the project.

35.3 EVALUATION OF COMMERCIAL FLAME RETARDANTS AND FINISHED FABRICS

The object of this part of the investigation was to evaluate the relative flame-retardant efficiencies of commercially developed fire retardants for treated fabrics. Individual reports are available on the comparative evaluation of CM, Bancroft fabric, phosphamates, Pollack fabric, Montgomery Bros. treated fabric, Chemical Warfare Service-Massachusetts Institute of Technology treated fabric, Flamex, Chemical Warfare Service gasproofed-flameproofed fabric, Ellicote P-1, Flamort T.C., Southern Regional Laboratory treated fabric.

In the case of each commercial flame retardant and finished fabric tested, conclusions and recommendations are presented.

35.4 THERMAL DECOMPOSITION OF FIRE RETARDANT MATERIALS

The mechanism of the decomposition of cellulose at high temperatures was studied and the influence of various retardant chemicals determined.

At elevated temperatures (500 C), cellulose decomposes into three main fractions:

1. A charred residue composed mainly of carbon.
2. A tarry distillate of a highly inflammable nature.
3. A volatile fraction composed of water and fixed gases.

In the presence of typical fire retardant chemicals, such as borate mixtures and phosphates, the course of the decomposition of cellulose is radically changed. The charred residue is increased, tarry products are reduced to a very low amount, and the water plus gas fraction is substantially increased.

These marked changes in the ratio of the three types of decomposition products take place upon the addition of up to approximately 5 per cent of the effective fire retardants. Further addition of retardants does not appreciably affect the course of the reaction.

35.5 VOLATILE DECOMPOSITION PRODUCTS FROM FABRICS

During the pyrolysis and controlled combustion of fabrics, the volatile products consist of:

1. A highly inflammable tarry distillate.
2. An aqueous fraction.
3. Smaller quantities of permanent gases consisting of water solubles (probably lower aldehydes and acids) CO, CO_2, and hydrocarbons.

Fire retardants markedly reduced the amount of tarry distillate. The amount of tarry distillate is directly related to the flaming tendencies of the fabric. The inflammable tar must be reduced below 2 mg per sq cm of fabric to provide adequate protection against flaming. Both retardants and non-retardants increased the amount of water and permanent gases, and changed the composition of these gases. There appears to be no definite relationship between these gases and the flaming tendencies of the fabric.

35.6 EFFECT OF TRACES OF FIRE RETARDANTS ON COTTON FABRICS

Traces of fire retardant chemicals such as CM or ammonium dihydrogen phosphate added directly to a fabric or remaining after leaching of an effectively treated fabric have the following effects on the flameproofing:

1. They may increase the rate at which a flame propagate along the fabric to a value equal to twice that at which an untreated fabric is consumed by the flame.
2. They may increase the duration of the afterglow to give glowing times approximately double those of untreated fabric.

Traces of retardants remaining in fabrics after the major amounts have been removed by water leaching have a definite detrimental effect on the fire resistance of the fabric. Less protection may result than that given by untreated fabric.

35.7 FUNDAMENTAL ASPECTS OF UREA-PHOSPHATE FLAMEPROOFING

Urea-phosphate flameproofing functions by the fixation of phosphate within the fabric as an ester of cellulose. Normally, this ester exists as the nitrogen salt of the unreacted phosphoric acid groups, and is capable of ion exchange. The exchange, principally with calcium and magnesium ions, results in loss of the flameproofed qualities of the fabric if the nitrogen-phosphoric acid content falls below a critical value.

The factors influencing the resistance to ion exchange are the original bath composition, the cure temperature, and the cure time. For permanence toward sea water and laundering, the most favorable conditions are high concentration of urea in the bath, high cure temperatures and long cure times. These conditions, presumably, convert the nitrogen salt to the nonexchangeable amide.

A formulation is recommended for flameproofing cotton fabrics to give sufficient permanence to withstand cold and hot water washing, 2 hours tumbling in sea water and six launderings with GI soap.

35.8 RATES OF DEGRADATION OF FLAMEPROOFED FABRICS

The thermal behavior of fabrics at high temperature indicates the following:

1. Fabrics decompose in two main stages, one associated with the disintegration of the cellulose, the other associated with the oxidation of the products of the primary dissociation.
2. These two stages coincide with the afterflaming and afterglowing tendencies of the fabrics.
3. Fire retardants decrease the thermal intensity of these two reactions as well as their rates.
4. The reaction most retarded corresponds with the afterflame or afterglow prevention qualities of the retardant, as measured by standard flame tests.

35.9 INSULATION VALUE OF FLAMEPROOFED FABRICS

The protection afforded by flameproofed fabrics during contacts with heat sources of high intensity was determined. It was found that untreated 8½-oz. herringbone twill offered appreciable protection against intense heat, provided the exposure is of short duration and does not raise the fabric to the ignition temperature. Herringbone twill, which is both adequately flameproofed and glowproofed, exhibits much greater insulation against heat or flame than a comparable fabric which is highly flame resistant, but does not possess sufficient resistance to afterglow. An efficiently flameproofed herringbone twill exhibits thermal insulation comparable to an asbestos sheet of approximately the same weight and thickness. Certain types of treated herringbone twill are equal or superior to Fiberglas asbestos or Fiberglas-Neoprene fabrics in their ability to insulate against heat or flame. This is true whether the fabrics are considered as single ply or two- or three-ply systems.

Fabrics which are highly flame resistant and also possess good resistance to afterglow offer a two-way protection against heat or flame. The wearer gains precious seconds of additional escape time by virtue of the low conductivity of the garment and, once removed from the source of heat, is not exposed to danger of burns from flaming or glowing clothing.

Military clothing, flameproofed by the UDAP or Bancroft type of treatment, appears to be the best available solution to the problem of protection against flame attack.

35.10 CHEMICAL AND PHYSICAL PROPERTIES OF FLAMEPROOFING AGENTS

The chemical and physical properties of the better flameproofing agents and mixtures were collected and tabulated. The data were available as an aid in the investigation of the mechanism of flameproofing.

The properties of the following simpler flameproofing agents were studied:

Borax.
Boric acid.
Borax: boric acid (7:3).
Borax: boric acid (1:1).
Borax: boric acid (3:7).
Borax: boric acid: diammonium hydrogen phosphate (7:3:5).
Ammonium sulfamate.
Ammonium dihydrogen phosphate.
Diammonium hydrogen phosphate.
du Pont CM.
du Pont CM — modified.
du Pont 3-WG.
du Pont T.
Ammonium chloride.
Ammonium bromide.
Ammonium iodide.
Urea.
Urea:phosphate (2:1 molar).
Urea:phosphate (4:1 molar).
Urea:phosphate (6:1 molar).
Urea:diammonium phosphate (4:4:1 molar)

The major portion of the experimental work was concerned with the measurement of *p*H of aqueous solutions, the solubility of flameproofing agents and their behavior on being heated up to temperatures approximating that of a flame. The heating characteristics were determined by visual heating experiments and more precise heating curve measurements.

35.11 KINETICS OF THE OXIDATION OF CARBON

Glow retardants appear to function by effecting the course of the oxidation of the chars remaining after the flaming reaction. The chars from glowproofed fabrics are preferentially oxidized to CO, rather than to CO_2.

The heat liberated in the reaction to CO appears to be insufficient to propagate a glowing reaction after the instigating source is removed. The reaction of the higher exothermicity (CO_2) appears to be the self-sustaining glow reaction. The mechanism whereby such reactions are favored by boric or phosphoric acid generating materials appears to be catalytic. The reaction is essentially independent of the catalyst concentration (above 1 per cent), and is readily capable of being poisoned by sodium salts. Sodium salts poison the boric acid catalyst more readily than they poison the phosphoric acid catalyst.

The tenacity with which the phosphoric and boric acids are held on the chars indicates that the mechanism is associated with a surface catalysis phenomenon. The data are insufficient to determine the type of sorption involved.

Chapter 36

ORGANIC COATINGS

36.1 SUMMARY

A research program was planned for the development of organic coatings for metals, which would be rust inhibiting, easy to apply, and produced without the use of any critical materials. Since at the time the tung oil supply was very critical, it seemed necessary, especially in the "Blitz" water can linings, to discontinue the use in such finishes. In addition, it seemed desirable to study new oils to determine whether they could replace tung oil in several types of finishes required.

A number of new oils were evaluated as tung oil substitutes in several types of finishes, especially in water can linings. They passed the standard hot and cold water tests, and many showed no softening or discoloration when removed from the water. By using suitable resins, good alkali resistance was obtained. Many of these oils may be substituted for tung oil without loss of serviceability characteristics.

In the study of air-drying food can coatings, finishes were developed which laboratory tests indicated are equal to the present type of finish in speed of drying and adhesion, and superior in durability and salt water resistance. No further conclusions can be made until large scale application and exposure tests have been made. Several of these finishes contained no alkyd resin.

Considerable work was completed on baked finishes for the exterior of food cans. Salt spray and weatherometer data indicated that they were satisfactory for durability and salt water resistance. Finishes tested for detail passed fabrication tests, indicating that they are entirely satisfactory for use under specification CQD-201A. None of the experimental finishes proved to be equal to the standard in processing tests; however, they were equal to the standard when applied at equal film weights. Some adjustment in pigmentation will need to be made so that experimental finishes will give satisfactory covering when applied at the same film thickness as the present material.

Based on laboratory prepared finishes, nine experimental water can linings were prepared on a commercial scale and tested by the Quartermaster Corps in accordance with specification JQD-111C. All except one of the experimental finishes, which were made with relatively available material, were found to be fully equivalent to the present tung oil type.

36.2 EVALUATION OF SPECIAL OILS AS SUBSTITUTES FOR TUNG OIL

Many oil companies have been actively engaged in developing oils which would give characteristics similar to those imparted by tung oil, including dehydrated castor oils, conjugated oils made from linseed oil or soybean oil, oils consisting of pentaerythritol, sorbitol, esters of fatty acids, and fractionated oils. The specific oils used were dehydrated castor oil, Kellin, du Pont GF-35 oil, Atlas Powder "K" oil, Roosenol 200, Zymol, Heyden 395 oil, Bakelite Modified linseed oil 1, Falkwood, Spencer-Kellogg Experimental oil XA-1, Conjulin, Spencer-Kellogg Experimental oil T-2, Roosenol 100, and Select Oil 200 (Pittsburgh Plate Glass Co.). These oils were combined with several types of resins to make varnishes of varying oil length. The following resins were used: unmodified phenol formaldehyde (*p*-phenylphenol) — Bakelite BR254, Beckacite 254, unmodified phenol formaldehyde (alkyl substituted) — Arofene 775, Bakelite 4036, rosin modified phenol formaldehyde — Amberol M-93, Arochem 365, maleic anhydride — Amberol 801, Arochem 605, Teglac Z-152, Pentalyn G, ester gum — Reichholdt Chemicals No. 1201, Congo, fused — No. 5, Strook & Wittenburg. Most of the varnishes were 20 to 25 gal in length.

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36.2.1 Twenty to Twenty-Five Gallon Varnishes

All varnishes passed the hot and cold water tests, many showing no effect when observed immediately after the films were removed from the water. Especially interesting, where extreme water resistance is desired, were Amberol M-93 with du Pont GF-35 oil, Bakelite BR254 with du Pont GF-35, Bakelite BR254 with modified linseed oil, Bakelite BR254 with Roosenol 200, Pentalyn G with du Pont GF-25, and Arochem 605 with Zymol. Finishes made with GF-35 and Falkwood were somewhat dark in color. The rate of bodying in the kettle was normal. The rate of bodying in the case of GF-35, Roosenol 200, and Conjulin were approximately the same as that of tung oil. Fast drying, comparable to the 4-hr type of tung oil coatings, was secured with practically all the oils. The following were of particular interest: Amberol M-93 with Roosenol 200, Bakelite BR254 with K oil, Pentalyn G with Kellin, Amberol 801 with Zymol, Bakelite BR254 with Zymol, Amberol M-93 with Conjulin, Teglac Z-152 with GF-35, Arochem 605 with T-2, and Pentalyn G with Heyden oil. A number of finishes made with these oils and with rosin modified phenol formaldehyde resins have good resistance to 2 per cent sodium hydroxide solutions; Amberol M-93 with Kellin, Teglac Z-152 with Roosenol 200, Arochem 605 with Roosenol 200, Amberol M-93 with T-2 were especially satisfactory. The finishes made with 100 per cent phenol formaldehyde resins gave excellent resistance to 5 per cent sodium hydroxide.

36.2.2 Thirty-Five Gallon Varnishes

All varnishes passed both hot and cold water test. Amberol M-93 with Zellin, Bakelite BR-254 with Kellin, Bakelite BR-254 with Zymol, Bakelite BR-254 with K oil, and ester gum with Zellin were of special interest. Good resistance to 5 per cent sodium hydroxide was obtained in some cases. The rate of bodying in the kettle was about normal for quick drying varnishes.

36.2.3 Fifty-Five Gallon Varnishes

These finishes were prepared with only a few of the newer oils to see if they would meet the requirements of specification TT-V-121a. Their rate of bodying was satisfactory. All samples dried hard in 24 hours, were tack-free in 18 hours, and passed the water-resistance tests. Amberol M-93 with GF-35, and ester gum with GF-35 showed good resistance to dilute alkali. It was concluded that most of the newer oils used with rosin modified phenolic or maleic resins will produce satisfactory finishes of this type.

Fast drying and satisfactory water-resistant finishes were made with Select oil 200 and Roosenol 100. Varnishes of good alkali resistance could be made with K oil, if the temperature were raised to 580 F. Finishes which were identical, except that in one case the top heat was 560 F and in the other 580 F, were compared. Those varnishes raised to 560 F gave only fair alkali resistance, while the others were excellent. The finishes made with Congo resin were dark in color but dried fairly rapidly. Fused Congo with GF-35, Fused Congo with Roosenol 200, and Fused Congo with T-2 set in 1½ hours and dried hard within 7 hours. All were satisfactory for water resistance, but poor in resistance to dilute alkali with the exception of Fused Congo with GF-35. Changes in driers used would give improved alkali resistance.

In general, fast drying varnishes were formulated with the oils under investigation. The type of resin affects the speed of drying and the time of cooking in the kettle. Such finishes have satisfactory water resistance, and by selecting the proper type of resin good alkali resistance was obtained.

These oils produced finishes which had most of the characteristics of tung oil varnishes. On account of the rate of bodying, the production per kettle will be very little less than with tung oil coatings.

36.3 EXTERIOR AIR-DRY COATINGS FOR FOOD CANS

Many types of quick drying materials were tested to develop a coating to meet requirements of CQD 200-A Type II, which would be superior to the present type of finish in speed of drying, ease of application, adhesion, flexibility, and durability, and which would contain no nitrocellulose (and preferably no alkyd resin). Those coatings which show promise were pigmented to olive-drab 319 of color card supplement to specification 3-1. Those which appeared satisfactory for speed of drying, appearance, and flexibility were applied to tin plate, and salt spray and weatherometer tests were pigmented to olive-drab 319 of color card XK-16700 and XK-16624 appeared promising, but in the early tests were difficult to pigment. A smooth enamel could be obtained when the pigment and the resin solution were ground together. Some blends of Cumar resins gave excellent results immediately after drying, but most became brittle after aging one week. None passed the weatherometer and salt spray tests.

Films made with Vinylite VYHH dried rapidly, but possessed poor adhesion and flexibility. Excellent results were secured if these finishes were applied over a suitable primer. Satisfactory results were obtained with two Acryloid finishes.

Several of the finishes examined appeared to be equal to the present type in speed of drying and adhesion, at least equally easy to apply, and superior in durability and salt water resistance.

36.4 EXTERIOR COATINGS FOR FOOD CANS

Oleoresinous varnishes were prepared using resins and oils which would be suitable for baking finishes. They were pigmented to meet the color requirements (olive-drab 319 of color card supplement to specification 3-1), applied to tin plates by roller coating, and tested for adhesion, flexibility, and water resistance. The only failures occurred at the double seaming of the can ends. This effect probably can be corrected by changes in pigmentation so that the film weights will correspond to those now in use. In the laboratory, where approximately equal film weights were used, the experimental finishes tested were at least as good as the standard in both fabrication and processing. All of these finishes passed fabrication tests. Several of the experimental finishes were almost as good as the present material and on some types of tin plate, some experimental finishes were rated higher than the standard.

36.5 INTERIOR COATING FOR WATER CANS

A finish to conform to specification JQD-111C should be reasonably light in color and have sufficient flexibility and hardness to withstand service conditions. It must satisfy specified resistance tests, impart no taste to coffee and lemonade, and be unaffected by a number of chemicals. It should also be applied by spraying and then baked at 375 to 425 F. Finishes were prepared, some containing varying amounts of melamine formaldehyde, pigmented, applied to metal containers, and tested. From the data obtained, it was concluded that the finishes made with the following oils are more suitable than the standard finish made with tung oil: XA-1, Zymol, Kellin, GF-35, and K oil. If more weight was given to the service test, Roosenol 200 was also better than tung oil. The development of interior coatings for food cans was considered, but no program was initiated.

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Chapter 37

COATED FABRICS AND THIN FILMS

37.1 WATER VAPOR PERMEABILITY OF PLASTIC FILMS

37.1.1 The Determination of Water Vapor Permeability

A PERMEABILITY RELATIONSHIP was established in terms of the solubility coefficient and diffusion constant. Experimental procedures for the evaluation of the permeability were discussed. Diagrams of a variety of permeability and diffusion cells are included. Values are given for the permeability constant, diffusion constant, and solubility coefficient for a wide variety of polymer films. Consideration was given to the effects of thickness, pressure, and plasticizers on the permeability.

37.1.2 Temperature Dependence of Water Vapor Permeability

The experimental results indicated that the temperature dependence of permeability was of such magnitude and varies so much from one film material to another that it was possible for one film to be a much better barrier at room temperature but quite the opposite at freezing temperatures. There are described a number of direct measurements of the influence of temperature on water vapor permeation for a number of self-supporting films.

37.1.3 Permeability Relation and the Effect of Plasticization

The rate of permeation of a gas through a polymer as a function of temperature may be represented as

$$P = P_o e^{-E/RT}$$

All available data on permeability of gasses through polymers showed that for a given gas there was a linear relationship between P_o and E (the energy of activation). An explanation was offered for this apparent relationship. The effect of plasticization on the permeation of water vapor was studied experimentally and it was shown that the lowering of the heat of solution was the predominant effect. From the data, the entropy of solution may be calculated and was interpreted as showing that water molecules, dissolved in polymer, exhibit much less freedom than when they are dissolved in plasticized polymer.

37.2 VISCOELASTIC PROPERTIES OF PLASTICIZED VINYL FILMS

37.2.1 Transition Phenomena in High Polymers

The significance of first order and second order transition points was considered. The effects of molecular weight, crosslinking, copolymerization, and plasticizer action on the apparent second order transition were discussed. The observed properties of an elastomer may be considered in terms of a molecular model in which there exists a range of internal and external Brownian movement. The internal Brownian movement or "local" fluidity is responsible for the contraction of the stretched material while the degree of external Brownian movement measures the degree to which the material will flow. Crosslinking procedures fixed strong chemical bonds between individual chains which allow for the rapid extension and contraction of the sample, if local viscosity is sufficiently low, while the ties between molecules prevent permanent flow in the sample as a whole.

37.2.2 Theoretical Discussion of Viscoelastic Behavior

A theoretical treatment of the nature of stress and strain and their resolution into volume and shear effects is presented. The response of a viscoelastic material to a simple shearing stress was considered in terms of a Maxwell and a Voight element. The simple stress considerations were generalized to include combined and inhomogeneous stresses.

37.2.3 Discussion and Illustration of Experimental Procedure

The experimental technique for the measurement of tensile creep properties of films was described. The principal limitations of this method were:

1. The lack of sensitivity of the method in the range of stiff materials.
2. Variations in film thickness.
3. Humidity control, found to be relatively unimportant for Vinylite films but extremely important for materials such as polyvinyl butyral.

A comparison was made of the effects of the plasticization of Vinylite VYNW with tricresyl phosphate and trioctyl phosphate. At short times the response of the latter was greater, while the former sample deformed to a greater extent in response to stresses of long duration. The activation energy of viscous behavior was determined to be 80,000 calories and above.

Curves of creep and recovery showed that within experimental error, the recovery curve may be predicted from a knowledge of the creep curve.

37.2.4 The Effect of Plasticizer Composition on Creep Characteristics of Vinylite VYNW

Plasticizers were divided roughly into four classes on the basis of the creep behavior which they impart to Vinylite VYNW:

1. Steep—high curves.
2. Steep—low curves.
3. Flat—high curves.
4. Flat—low curves.

The presence of ring structures in the plasticizer (i.e., tricresyl phosphate) produced curves of types 1 and 2. Plasticizers which sweat out belong usually to group 4.

For a plasticizer made up of trioctyl and tricresyl phosphates, the creep curve at low temperatures was between those of the pure plasticizers but at an intermediate temperature the mixture produces the more flexible film. Two generalizations which may be made with some reservation are:

1. That extreme softness or rubberiness may be obtained by the use of plasticizers such as trioctyl phosphate.
2. A moderate degree of flexibility existing over a wide range of temperature may be obtained by the use of a mixture of solvent and nonsolvent (PD-16)-type plasticizers.

Plasticizing action was considered as a dynamic process of diffusion of plasticizer, which by migration causes the breaking and reforming of polymer-polymer contacts.

37.2.5 The Effect of Resin Composition on Creep Characteristics

Incorporation of Vinylite VYHH or VYNS into VYNW produced softer films at the expense of flatness of the creep curve. Plasticized polyvinyl butyral was softer than Vinylite VYNW with the same amount of plasticizer. This softness was attained at the expense of the shape of the creep curve and the temperature dependence of the stiffness.

Plasticization produced more than a lowering of the softening temperature of the Vinylite. The distribution of elastic relaxation was shifted to faster times and was spread out over a wider range of log *t*. In the case of nonpolar polymers, only the softening temperature was lowered.

Concentrated solutions of Vinylite VYNW in a poor solvent (methyl ethyl ketone) gave greater viscosity values than solutions in a good solvent (cyclohexanone), due to molecular clustering in the poor solvent. The same general effects were observed in plasticizer-Vinylite solutions.

37.3 SOLUTION PROPERTIES OF VINYL RESINS

37.3.1 Theory of Polymer-Liquid Systems

A theoretical treatment of polymer-liquid systems including recent extensions and modifications concerning the entropy of dilution was presented. A molecular theory was presented for ΔH_1, the heat of dilution. Solubility con-

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siderations were extended to include the recent theories of fractionation. The importance of the compatibility constant was discussed.

37.3.2 Fractionation and Distribution Curves of Vinyl Resins

A detailed discussion was presented of the various experimental techniques of fractionation used. It was determined that while fractional extraction procedures gave satisfactory resolution in the high molecular weight range and poor resolution in the low, the reverse was true of the fractional precipitation method.

Distribution curves of Vinylite VYNW and Geon 101, as determined by the several methods, were presented. The distribution curve for both these polymers was sharp with a long low molecular weight tail. The number average molecular weights were reported as 113,000 for Vinylite VYNW and 150,000 for Geon 101.

37.3.3 Molecular Weight Technique

Osmotic Pressure

The report contains a brief summary of the theory of osmotic pressure. A complete discussion of the preparation and properties of denitrated nitrocellulose and cellophane membranes is followed by a description of the construction and operation of the three types of osmometers used.

Data are given for the number average molecular weights for the various Vinylite and Geon resins, along with the molecular weight of several fractions. The molecular weight range of the Vinylite resins covers from 36,500 for VYLF to 133,000 for Vinylite VYNW 998. Geon 101 and 202 have molecular weights of 115,000 and 120,000, respectively.

The solvent action, as determined from the slope of the π/cc plot gives this order: Dioxane (poor), methyl ethyl ketone (fair), and cyclohexanone (good).

Light Scattering

The theory of the light-scattering technique and the experimental details are discussed briefly.

Since the light scattering method was especially sensitive to the state of molecular aggregation in the solution being measured, serious difficulties are encountered in the measurement of the molecular weight of Geon 101 and its resins.

Values for the molecular weight of several Vinylite resins are given. Measurements of the dissymmetry of several of these solutions indicate that the polyvinyl chloride chains are much more extended than they would be if there were unhindered free rotation about the carbon-carbon bonds.

Viscosity

A brief treatment of the theory of viscosity is presented. The k' of the Huggins equation, on the basis of the experimental results, is interpreted as a constant whose magnitude depends, at least in part, on the number and permanence of contacts between adjacent solute molecules in solution, and its value is expected to decrease with increasing solvent action.

Data are given for the intrinsic viscosity k' and vinyl acetate content for the several polyvinyl chloride-polyvinyl acetate polymers investigated.

The relation between intrinsic viscosity and molecular weight for Vinylite VYNW fractions over the molecular weight range studied may be expressed as

$$(\eta) = KM^a$$

where

$$K = 3.16 \times 10^{-6}$$
$$a = 1.1$$

Association of Polyvinyl Chloride in Solution

Measurement of the molecular weight of a Geon 101 fraction in dioxane, by the osmotic pressure technique, produced the values in the following table:

Temperature, C	Molecular Weight
14	210,000
38	210,000
47	141,000
68	112,000
77	90,000

Osmotic pressure data for the same fraction in cyclohexanone gave molecular weights between 94,000 and 98,000. The change of molecular weight with temperature in dioxane solution was indicative of an association-disassociation phenomenon.

Similar effects are to be observed by light-scattering techniques for the Geon fraction in dioxane or in cyclohexanone to which nonsolvent has been added.

Examination of the Geon-dioxane system in the ultracentrifuge shows the presence of two components. The quantity of the heavier component decreases while that of the lighter component increases on heating.

It appears then that a large portion of the polyvinyl chloride in Geon 101 has the ability to form associated clusters when dissolved in dioxane or in a similarly poor solvent.

37.3.4 The Determination of Polymer-Liquid Interaction by Swelling Measurements

For a large number of polymer-liquid systems μ values have been determined by the application of the Flory-Rehner equation to the equilibrium swelling values of crosslinked polyvinyl chloride film. Data are given for polyvinyl chloride in a large number of liquids, with μ values ranging from 2.8 to -0.85.

According to the data, μ was found to be temperature independent in the region 0.2 to 0.4. Investigation of osmotic pressure data indicated the same boundary values for indifferent solvents. For μ values less than 0.3, $\Delta\bar{H}$ must be negative indicating strong polymer-solvent interaction. If μ becomes more positive (0.3 to 0.55), polymer-polymer contacts are preferred. Plasticizers with μ values greater than 0.55 "sweat out."

With increasing temperature μ varied in such a manner as to approach a value between 0.2 and 0.4.

37.3.5 The Solubility of Polyvinyl Chloride Resins

The minimum gel concentrations of Vinylite VYNW have been determined in a wide variety of solvents. The cohesive energy density of hydrocarbons and halogenated hydrocarbons was in the neighborhood of 90 cal/cc. Since the μ value of these systems was small, it is estimated that the cohesive energy density of polyvinyl chloride acetate was about 90 cal/cc. Using this value, other solvent systems were investigated. Ketones, esters, and nitro compounds were found to be active solvents, the specific forces probably being hydrogen bonds between the hydrogens on the chlorine carrying carbon and the negative oxygens of the solvent.

For homologous series of plasticizers μ values have been determined. It appears that μ falls from a high value as molecular weight of solvent increases in a given series and then rises again.

Dilution ratio measurements have been compared with μ values. For a series of comparable compounds the dilution ratio was found to increase as μ decreases.

37.4 HEAT AND LIGHT AGING OF HIGH POLYMERS

37.4.1 Theory of Aging

A consideration of the elementary processes which occur during the aging of polymers was presented, along with chemical methods for the determination of chemical groups in polymers.

The use of the infrared absorption spectrum to investigate the chemical changes during aging was presented along with a table of structural data for a variety of polymers. Consideration was given to the kinetics of oxygen absorption.

37.4.2 Degradation of Vinyl and Diene Polymers

Studies on simultaneous polymerization and degradation showed that a steady-state viscosity results in the presence of benzoyl peroxide. It follows that catalysts which decompose into free radicals may also catalyze degradation. This effect has been observed for polystyrene and methyl acrylate systems.

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Factors affecting the rate of oxidation were:

1. Chemical structure. The double bond and methyl group of natural rubber enhance the rate of oxygen absorption. Replacement of the methyl group with Cl, CN, and amide, etc., resulted in a retardation of the rate of oxidation.

2. Antioxidants. The initial rate of oxygen pickup was greatest in a stock containing benzoyl peroxide and slowest in a stock containing phenyl β-naphthylamine. The rate of stress increase was most rapid in the former showing that crosslinking and scission are accelerated by benzoyl peroxide and retarded by phenyl β-naphthylamine.

3. Vulcanization. Sulfurless hevea showed at least a sevenfold retardation in oxygen absorption when compared to a similar sulfur cured stock.

37.4.3 Evaluation of Light Sources

It was determined that the rate of aging is dependent upon:

1. The wavelength distribution of the intensity.
2. The wavelength sensitivity of the polymer.

Since it is believed that the existing discrepancies between sunlight-aged and artificially aged materials are due largely to variations in the characteristics of the light sources, an investigation of those sources was made. Comparisons are given between sunlight, National Fading Unit, and Atlas Fadeometer on the basis of the spectral intensity distribution.

A sample calculation was included for the determination of the relative intensities of the different filter combinations. From these the total intensity over the entire spectrum may be calculated.

37.4.4 Heat and Light Aging of Polyvinyl Chloride

Polyvinyl chloride under heat releases HCl gas. The accumulation of HCl results in an accelerated decomposition of the film. The presence of oxygen did not appear to affect the rate of HCl release.

Color changes on aging were attributed to an increase in the number and in the length of conjugated systems of double bonds. Systems containing more than about five double bonds have a reddish color similar to that of aged polyvinyl chloride.

The effects of a stabilizer appeared to be negative since it does not combine with the liberated HCl and yet depressed the evolution rate below that of the unstabilized film when HCl was not accumulating.

The effect of ultraviolet light was to decrease the molecular weight of the resins. However, films exposed for elongated time periods showed a crosslinking effect. The presence of plasticizer reduced the amount of crosslinking without affecting the rate of chain scission markedly.

It was found that wavelengths below 350 $m\mu$ are mainly responsible for breakdown.

Exposure to ultraviolet light sensitized polyvinyl chloride so that subsequent heating caused a marked breakdown.

37.4.5 Light Aging of Synthetic Rubbers

Outdoor exposure of rubber products caused degradation by three mechanisms: (1) ozone attack, (2) thermal oxidation, and (3) photoactivated oxidation.

Elongation or flexure of samples was important in ozone aging. Temperature had a very secondary effect. Ozone aging was a vital factor in the aging of natural and butadiene copolymer rubbers. It was of secondary importance for butyl, Neoprene, and polysulfide rubbers. Thermal oxidation in the absence of light was of importance for all rubber products used at elevated temperatures or whose use produced high temperatures. Infrared from the sun or from artificial sources caused appreciable surface heating which accelerated oxidation. Photo-oxidation sometimes retarded ozone attack by forming an impervious surface skin.

Many fillers, particularly carbon black, absorb most of the active radiation and effectively retard photo-sensitization. Many antioxidants which retard oxidation accelerate photo-oxidation.

The effect of light appeared to be the acceleration of oxidation without changing the nature of the aging process. Butyl rubber softens, and GR-S hardens, both by heat and light aging. The combined effect of both heat and light was greater than the sum of the individual effects.

A rough proportionality existed between the total quantity of radiation and the extent of degradation.

37.5 MECHANICAL PROPERTIES OF FILMS AND COATED FABRICS

37.5.1 The Relationship of Mechanical Properties to Molecular Chain Lengths in Vinyl Polymers

Fractions of Vinylite VYNW from the higher molecular weight range (upper 88 per cent of distribution curve) had an average folding endurance of 3,900 MIT double folds. This value was 90 per cent higher than the folding endurance for unfractionated Vinylite VYNW. The low molecular weight fractions had values of 510 to 610. The folding endurance becomes independent of molecular weight at about 130,000. Tensile strength falls off sharply as molecular weight decreases below 80,000.

Measurement of folding endurance and tensile strength for leached and vacuum dried films gave higher values of the former for leached and higher values of the latter for vacuum dried. It was evident that the method of preparation had a significant bearing on the mechanical properties.

37.5.2 Development of Films for Quartermaster Uses

This section included an enumeration of the properties most desired in a film as well as the characteristics of a number of commercially prepared films. Tables of strength values of the differently prepared vinyl and polyethylene films were given along with aging and low-temperature characteristics.

Saran-coated cellophane was found to be a superior packaging material for the wrapping of moisture containing materials. The loss of moisture, by weight, of the product wrapped in Saran-coated cellophane was shown to be far less than that of the same product wrapped in moistureproof cellophane.

The methods of preparation of films on a laboratory scale are described in sufficient detail. Methods for the casting of plasticized and unplasticized vinyl films and rubber films are given.

37.5.3 Developments in Coated Fabrics for Quartermaster Uses

This section was concerned primarily with a discussion of the test data obtained in the evaluation program on poncho and raincoat-type materials. Tables of test data for alkyd modified oil-coated airplane cloth, polyvinyl butyral-coated fabrics, vinyl-coated nylon and cotton, etc., are given along with the discussion.

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Chapter 38

WEAR RESISTANCE OF APPAREL TEXTILES

38.1 INTRODUCTION

THE OBJECTS of this investigation of wear resistance of apparel textiles were: (1) to design a classification, scoring, and evaluation method whereby apparel textiles subjected to the Camp Lee Combat Course may be ranked for their wear resistance; (2) to study laboratory abrasion-testing machines and techniques and develop laboratory criteria for ranking the abrasion-resisting properties of fabrics; (3) to correlate Camp Lee Combat Course results with laboratory abrasion-testing methods; and (4) to recommend improvements in fabric construction and/or finish which will increase the abrasion resistance of textiles in service.

The Combat Course at Camp Lee, Virginia, was used to test the utility of military clothing and equipage under controlled and reproducible service conditions. The course consists of fences, road blocks, gravel roads, shell holes, concrete culverts, bombed out houses, rubble, etc., arranged so that the soldier passes through the obstacles chronologically and in a prescribed manner.

38.2 TEST PROCEDURE

This study was concerned with the wear resistance of apparel textiles tested upon the course and methods of correlating these results with abrasion resistance determined upon conventional laboratory abrasion-testing machines. Three cotton fabrics were studied: (1) 8.2-oz twill, (2) 9-oz sateen, and (3) 9.3-oz herringbone twill [HBT]. Fifteen uniforms, i.e., a jacket and pair of trousers, were prepared from each fabric for the Combat Course test. All garments were made with warp yarns running lengthwise. The twill and HBT were prepared face outward, the sateen back outward. Each soldier taking part in the test had one uniform of each fabric with which he made 15 Combat Course traversals, wearing each fabric in turn. At the end of each traversal, the garments were inspected and all damages recorded. They were then subjected to five more cycles. Analysis of the data was, however, made only through the first fifteen cycles, since this was sufficient to determine fabric differences.

38.3 COMBAT COURSE RESULTS

Preliminary study of the fabrics subjected to the course showed the existence of fundamental damage classes. (1) Wear areas: mild, medium, and severe; (2) Holes; (3) Holes in wear; and (4) Tears (considered a special type of hole). The size of each individual damage was calculated as the area of a rectangle of length and width equal to the maximum and minimum diameter of the damage area.

By summing up areas of the same damage class, the Net Surface Damage [S] in square inches for each case was recorded at each cycle. A graph of S versus cycles was plotted, and the destructive index was calculated from this graph. To state that an end point alone expressed in Combat Course cycles to produce any selected degree of destruction was a valid criterion of "wear resistance" is to neglect the importance of rate of destruction. Rate of destruction essentially measured the changing condition of the garment as it proceeds to destruction as a result of abrasion. The destructive index was a number indicating the rate of damage to any garment at any cycle, taking into account the entire past history of the garment. The lower the destructive index, the better the garment. The destructive indices for each damage class for each fabric were calculated at each cycle. A combined destructive index for all damage classes was calculated by weighting S for each damage class by its per cent reduction in warp tensile strength, summing the weighted S, and calculating a destructive index.

Combat Course destructive indices showed: (1) the sateen wore out most slowly, and was therefore best; (2) herringtone twill wore 1.8 times faster than the sateen, and was next best; (3) the twill wore out 2.1 times faster than the sateen, and was the poorest; (4) the twill probably wore out slightly faster than the herringbone twill (the difference between them was small compared with differences between twill and sateen and between herringbone twill and sateen). These conclusions apply to the twill face, herringbone twill face, and sateen back when the warp yarns run longitudinally in the garment.

38.4 LABORATORY TEST RESULTS

The Massachusetts Institute of Technology unidirectional and the Taber multidirectional abrasion machines were studied in the laboratory. Samples of the three fabrics were abraded for a varying number of cycles, and the tensile strength of the abraded portions was determined. Graphs of per cent loss in tensile strength versus abrasion cycles were then plotted for the following abrasion and test directions for each fabric, face and back, for each of the two machines: (1) abrade warp-test warp, (2) abrade filling-test warp, (3) abrade filling-test filling, and (4) abrade warp-test filling. Areas under the per cent loss in strength versus cycles curve are laboratory destructive indices and may be compared with Combat Course destructive index values.

Both the Massachusetts Institute of Technology and the Taber machine ranked the abrasion resistance of the fabrics in substantially the same order. Both show per cent loss in strength versus cycles curves of the same mathematical type, namely, $C - e^{KL}$, where C is abrasion cycles, e the natural logarithm base, L the per cent loss in strength, and K a constant which varies with fabric geometry, abrasion direction, and tensile strength direction. Therefore, no direct relationship between the Taber and the Massachusetts Institute of Technology machine is known at present.

Laboratory results indicated that fabrics abraded in different directions show the same resistance rank when tested in a single direction; fabrics abraded in a single direction show inverse abrasion resistance rank when tested in perpendicular directions. The sateen back when abraded and tested warpwise, and the twill face when abraded and tested fillingwise, were the most abrasion-resisting surfaces.

The inherent abrasion resistances of the three fabrics were identical in that they were all made of cotton. Differences between the six surfaces were differences of fabric geometry, e.g., yarn size, twist, sett, diameter, fabric weave, picks and ends per inch, per cent warp or filling yarns on the fabric surface, and float length. Such noninherent fiber properties were collectively called form factor. This procedure controlled the abrasion resistance of fabrics made from the same fiber. A fabric surface which had high abrasion resistance in one abrasion and test direction had poor abrasion resistance in the perpendicular abrasion and test direction. If the six surfaces were abraded and tensioned equally in all directions, they should have equal abrasion resistances. This was demonstrated by summing the laboratory destructive indices for the four abrasion and test directions. There resulted no significant difference in abrasion resistance rank between the six surfaces.

The fabrics had specific abrasion resistances for specific abrasion and test directions. To predict the abrasion resistance of a fabric under service conditions, the predominant abrasion and tension directions must be known. Photomacrographs (19×) of each fabric were prepared to assist in interpreting the laboratory abrasion results, Combat Course results, and the correlation between them.

Analysis of the motion of the men as they traversed the course indicated that the direction of abrasion and tension was predominantly longitudinal to the man. Since the garments were made with the warp yarns running lengthwise, it was predicted that the best course fabric would be that which was best able particularly to withstand warpwise abrasion and tension. This prediction was confirmed and explained by photomacrographs. The twill and herringbone twill face fabrics are warp flush. Warpwise abrasion during the course traversal caused severe warp yarn damage, resulting in warp failures under tension. The sateen back was a filling flush fabric; warp yarns under the same abrasion and tension conditions were protected.

Similarly, the laboratory abrasion resistance of every fabric surface for every abrasion and test direction may be predicted from photomacrographs. Correlation between the prediction of Massachusetts Institute of Technology and Taber results was excellent. Course results show that the twill face and herringbone twill face have approximately twice the destructive index of the sateen back. For quantitative correlation, laboratory destructive indices should bear this same relation. If abrade warp-test warp laboratory destructive indices are compared, twill face-sateen back and herringbone twill face-sateen back ratios were much higher than the required 2:1. Therefore, other abrasion and test directions must be considered to reduce these ratios until they become equivalent to course ratios. To arrive at proper ratios, empirically selected percentages of the various laboratory abrasion and test directions must be combined to form a weighted laboratory destructive index. Experimentally, it was found that if the following percentages of laboratory destructive indices for the four abrasion and test directions are calculated and summed, the resulting combined indices for the twill face, sateen back, and herringbone twill face are in approximately the same ratios in the laboratory as they are on the course: abrade warp-test warp, 70 per cent; abrade filling-test warp, 5 per cent; abrade filling-test filling, 20 per cent; and abrade warp-test filling, 5 per cent.

These weighting factors apply only when the warp yarn runs lengthwise with the garment. Combat Course wear resistance of textile fabrics may be predicted from laboratory criteria by the following method: If the warp yarns run longitudinally with the garment, sum the following destructive indices: abrade warp-test warp, 70 per cent; abrade filling-test warp, 5 per cent; abrade filling-test filling, 20 per cent; abrade warp-test filling, 5 per cent. If the filling yarns run longitudinally with the garment, sum the destructive indices: abrade warp-test warp, 20 per cent; abrade filling-test warp, 5 per cent; abrade filling-test filling, 70 per cent; abrade warp-test filling, 5 per cent. In both cases, the lower the resulting number, the better the abrasion resistance and the better the garment will wear on the course.

38.5 CONCLUSIONS

On the basis of percentages, the following conclusions and predictions were made: If the garment is constructed with the warp yarns running longitudinally, the sateen back will be best under Combat Course test; also if the garment is constructed with the filling yarns running longitudinally, the twill face will be best.

Weighting values were empirically determined after extensive study of the Combat Course and apply only to the course. It is neither stated nor implied that wear resistance under any and all service conditions may be predicted by the use of such weighting values. To predict wear resistance under service conditions from laboratory methods, a knowledge of abrasion and tension components for such conditions is necessary. Weighting values may or may not be the same as those found empirically for the course. The use to which the garment is put would decide whether Combat Course values might judiciously be applied.

It is believed that, as a result of this investigation, laboratory methods can now, with reasonable accuracy, predict the serviceability of fabrics with a minimum of effort and time, compared with the length and complexity of a Combat Course test. The Combat Course, however, does more than evaluate fabrics. It judges clothing design and construction, utility of equipage, and aids in the solution of problems pertaining to comfort and convenience. It is doubtful whether laboratory techniques could ever solve such problems.

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Chapter 39

SHRINKPROOFING OF WOOL KNITTED ITEMS AND FABRICS

39.1 INTRODUCTION

THE TENDENCY of wool to felt under ordinary conditions of laundering is its outstanding disadvantage as a textile fiber, especially when used in such items of clothing as socks, underwear, shirts, and sweaters. In normal civilian uses, this disadvantage is in part overcome by extremely mild laundry conditions such as low temperatures and a minimum amount of mechanical action, conditions which are not obtainable under war operations. The military requirements for wool clothing are high, but these requirements have been greatly increased because of excessive shrinkage of these materials during field laundering.

The research program on shrink-resistant treatments for wool was undertaken to overcome this difficulty. Shrink-resistant processes have been known for many years but, for a variety of reasons, have never achieved any appreciable success in the United States. Most of the known processes are included in the following main types: (1) halogenation processes; (2) resin processes; (3) solvent-alkali processes; and (4) enzyme processes.

The cushion sole sock was considered most critical and accordingly the problem of imparting a shrink-resistant finish to this item was attacked first. The urgency of this problem made it necessary to utilize or develop processes which could be applied on equipment available in hosiery mills producing socks for military use. For this reason, considerable emphasis was placed on the halogenation processes, although a number of the resin processes as well as the alkali-solvent process showed considerable promise. Of the last two, the former required special curing equipment and the latter a solvent recovery system, neither of which were available in hosiery mills.

39.2 TEST METHODS

It was well known that all the halogenation processes result in a certain amount of damage to the fiber; it therefore became necessary to have tests for measuring both shrinkage and fiber damage in order to determine the point at which the benefits of shrinkage resistance are offset by the disadvantages of reduced strength and elasticity which result from fiber damage.

Test methods were developed for the following: measurement of shrinkage, laundry test, loss in weight, alkali solubility, 30 per cent index. This last measurement affords a very sensitive method for detecting changes in the fiber, but it does not tell what chemical reaction has occurred nor does it predict wearing qualities. These factors must be determined by correlation with other tests.

39.3 INVESTIGATION OF SHRINK-RESISTANT PROCESSES

In order to minimize the time interval between laboratory developments and practical application of such developments, a pilot plant was established. With these facilities it was possible to treat small commercial batches of socks under carefully controlled conditions.

39.3.1 Halogenation Processes

An investigation of various modifications of the halogenation processes was first undertaken. The processes investigated included: Hypol, Althouse, Lana-Seal, Neutronyx, Westvaco, Alrose, dry chlorination, alkaline hypochlorite, and acid hypochlorite. The Hypol process was developed and adopted as the procedure recommended for mill trials on the cushion sole socks.

39.3.2 Resin Processes

The resin processes investigated were as follows: Resloom, Lanaset, Dura-Lana. Although the results obtained with these resin processes were interesting, they were not recommended because of certain disadvantages in the industry such as lack of curing equipment.

39.3.3 Solvent-Alkali Process

The Freney-Lipson process was investigated. The shrinkage control was very good and the damage was not excessive except at high temperatures and large moisture contents.

39.3.4 Enzyme Process

The only process investigated was the Ficin process, applied to wool after a pretreatment with sodium bisulfite. Socks treated by this process showed a large amount of fiber damage, and also shrank considerably.

39.4 WEAR TESTS AT CAMP LEE

In order to correlate laboratory tests with actual performance tests, socks were worn by men at Camp Lee. Some of these were deliberately overtreated in order to estimate what limits might prove practical.

39.5 FUNDAMENTAL RESEARCHES

The experiments were concerned with the practical aspects of treating wool in order to reduce its tendency to felt and shrink. Simultaneously, some work of a more fundamental nature was conducted.

When the effect of *p*H was investigated, it was found that the shrinkage during laundering decreased with decreasing *p*H of treatment.

A number of preliminary experiments indicated that the reaction of the fiber with chlorine was influenced by the grade of wool (i.e., fiber diameter). In other words, in a given period of time, the finer fibers were affected to a greater degree than the coarser ones. The results obtained indicated a direct relationship between 30 per cent index and fiber diameter.

The idea has frequently been advanced that the nonshrinking characteristics of chlorinated wool are due to the fact that the scales are removed during chlorination treatments. Presumably the fiber becomes smooth and will not migrate in the fabric. The effect of the treatments on surface structure was investigated on fiber treated in the *p*H series conducted at the pilot plant. The conclusion was reached that modification of the scale structure is related to shrink resistance.

The fact that certain treatments appeared to remove or damage the scales suggested, but did not prove, that the treated fiber was more slippery than the untreated fiber. In order to test this idea, a special instrument was designed for measuring the coefficient of friction of a single fiber against a wool felt or other surfaces. All values for the coefficient of friction for treated fibers were higher than those for the corresponding untreated fibers within the limits of experimental error. The values measured dry did not change with *p*H of chlorination treatment, but those measured wet increased with decreasing *p*H and the low and high values tended to approach each other. This result means that not only is it more difficult to start the fibers in motion, but that they start as easily in one direction as in the other, thus eliminating any directional effect. The frictional properties are apparently important in the felting of wool.

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Chapter 40

PACKAGING WITH PLASTICS

40.1 INTRODUCTION

VARIOUS PLASTICS have been utilized in the form of thin film sheeting as wraps and bags during the past ten years. The recent developments in this field have produced packages exhibiting improved functional characteristics, but in general the limitations applying to wraps and bags made of thin films remain. In view of the fact that a considerable number of industrial organizations have developed technical skill in the handling of carton dipping processes, it appeared logical to seek further improvement of military packaging through the application of plastic materials in place of petroleum waxes. It was also known that there were many new chemical compounds under development in the laboratories of organizations producing plastics, and that some of these new materials might have the characteristics desired for improving military packaging.

The preliminary investigation under this project involved a survey of dipping compositions for application to packaging of rations. The objectives were as follows:

1. To obtain available dipping formulations (products) from commercial sources.
2. To set up a series of the most significant testing procedures for the relative evaluation of the above formulations.
3. To evaluate the materials under (1) by the testing procedures established under (2), observing both the interesting and unsatisfactory characteristics. Among the properties evaluated were:
    a. Temperature of application and viscosity at application temperature.
    b. Blocking behavior at 140 F.
    c. The effect of handling at −20 F on waterproofing.
    d. Water-vapor transmission.
    e. Odor and taste.
    f. Stability at application temperature.

The desirable properties of the finished package are as follows.

1. The material must be odorless, tasteless, and nontoxic. In the process of application, no undesirable odor or flavor should be transmitted to the contents of the package.
2. A package dipped in the plastic should withstand storage at a temperature of 140 F, without blocking and without the liberation of solvents to contaminate the contents.
3. The coating must be capable of withstanding rough handling at −20 F, without cracking, chipping, separation from the base paper, or loss of waterproofing.
4. The coating should be impervious to the penetration of grease or oil.
5. Water-vapor transmission rates measured in the General Foods Moisture-Vapor Transmission Cabinet should not exceed 0.1 gram per 100 sq in. per 24 hours. Materials with values up to 0.25 (comparable to those now in use) are of interest but substantial improvement is desirable.
6. The film should have good bursting strength, good tensile strength, and good resistance to tearing and abrasion.
7. The coating must be waterproof at the temperature extremes mentioned and be stable to both fresh and salt water.
8. The film should be impervious to the gases ordinarily employed in chemical warfare.
9. The coating must have good resistance to the penetration of insects and it is desirable that it be unattractive to rodents.
10. The film must be stable under field storage conditions for at least 24 months.
11. The coating should be transparent or sufficiently translucent to permit printing on the paperboard to be reasonably legible after application.
12. The coating should not support the growth of fungi.
13. The finished surface should preferably have a dull finish. Surfaces capable of reflecting light must be avoided.

14. The coating should preferably be noncombustible, but slow-burning types are not prohibited.

15. The film should be as nearly neutral as possible to avoid danger of corroding metallic containers used for ration components.

Dipping or completely immersing a filled and sealed package in a liquid appears to be the least complex method of applying a continuous film. In view of the comparative simplicity of this procedure and the backlog of technical skill developed by various organizations in the handling of carton dipping, application by this method at speeds of 60 cartons a minute appeared to be the preferred approach. However, other methods of application are not excluded if their advantages justify modifications of technique.

On the basis of use in a dipping process with a minimum change in existing procedures, the following properties should be considered:

1. An application temperature of between 180 and 210 F is desirable. One limiting factor here is the difficulty of obtaining bubble-free films at higher temperatures without predrying the paperboard. Another factor is the nature of the contents of the packages, which will include biscuits, caramels, fruit bars, and chocolate.
2. At the application temperature, the viscosity should be sufficiently low to permit draining of the excess material from the package.
3. The material should be stable for periods up to two weeks at application temperatures.
4. The coating should flow smoothly and bridge minor openings in the cartons.
5. Wax compounds costing up to 18½ cents per pound have been used. Allowable costs of other materials will vary depending on the amount required, advantages gained, and simplicity of operation.

Sixty-six commercial samples were received from industry as a result of a general request sent from the project. Twelve samples were believed suitable for test in the initial phase (hot melts) and forty-eight appeared interesting for compounding in the development phase. Six samples did not appear usable without some modification, such as the use of solvents or predrying of the cartons.

40.2 DEVELOPMENT OF TEST METHODS

40.2.1 Relations of Viscosity to Temperature

One of the most significant properties in the preliminary evaluation of dipping materials is the relation of temperature to viscosity. For standard dipping techniques, a maximum temperature of approximately 220 F is allowed. Above this temperature, moisture in the carton will be removed, partially destroying the continuity of the film. If a material is not liquid and sufficiently fluid at 220 F to permit dipping, special techniques would have to be employed, such as predrying of the cartons and the use of higher dipping temperatures. These modifications are possible if justified by other advantages. The development of special dipping techniques was considered to meet unusual requirements.

A method employing the Brookfield Synchrolectric viscosimeter was adopted for measuring temperature-viscosity relationships. This instrument utilizes a rotating spindle driven by a constant speed motor. Viscosities were measured by the drag on the spindle and were indicated directly in centipoises. By the use of four different spindles and four different speeds, full-scale readings from 100 to 100,000 centipoises were measured.

40.2.2 Blocking

The blocking test consisted of a test on a coated piece of carton stock, subjected to a pressure of 1 psi for 24 hours at temperatures of 100 and 140 F. Nonblocking at 140 F is desired; the test at 100 F is included to allow more flexibility and an estimate of possible improvement by modifications in the development phase of the program.

40.2.3 The Effect of Handling at —20F on Waterproofing

Satisfactory moistureproofing of a coated carton cannot be obtained unless the film of wax or resin is continuous and unbroken. An unfortunate characteristic of many coating materials is brittleness at low temperatures. Mechanical handling at such temperatures (produced by refrigeration, high altitude flight, or frigid weather) is likely to break a wax film. Subsequent exposure to atmospheric conditions of temperature and relative humidity then results in an undesirably high pickup of moisture.

Direct evaluations of resistance to such treatment involve numerous variables of application and handling, and are valid only for the conditions used. It was felt that a fundamental evaluation of the properties affecting such behavior would be preferable to an arbitrary comparison of coated cartons.

Among the factors governing the low-temperature behavior of a coated board are the following.

1. The properties of the board.
2. The anchorage of the wax or resin to the board, influenced by the viscosity of the wax, permeability and wettability of the board, and time of immersion.
3. The low-temperature flexibility of the wax or resin film.
4. The thickness of the film.

Undoubtedly, the most important factor is the low-temperature flexibility. If the coating material is sufficiently flexible, stresses resulting from differential contraction or from bending will not cause fracture. On the other hand, if the coating is brittle, only a small thickness can be used without danger of fracture. All other factors being the same, a more flexible material can be employed in a greater thickness and, hence, will afford better protection against moisture transfer.

The stresses which might break the wax film on a carton during shipment or handling are those associated with an object falling on, or against, the carton, or with the carton falling upon some object. Because there is reason to believe that flexibility of materials like waxes depends upon the rate of application of stress, static flexure tests are less suitable than dynamic measurements. In view of this consideration, a method of measurement of flexibility was desired in which the stress is built up very quickly.

The apparatus consists essentially of a pendulum which is freely suspended from a simple pin bearing (not lubricated). A clamp at the lower end of the pendulum grips one end of a strip of the wax under test. The pendulum is "cocked" at a rather large angle. After the specimen has been at the given low temperature for a time sufficient to be in equilibrium with the surroundings (the instrument is in an electric cold box), the pendulum is released. At the bottom of the swing, where the pendulum has its greatest speed, the lower end of the specimen meets a fixed anvil. The specimen strip then bends and breaks, the degree of bend to rupture depending upon the flexibility of the wax.

40.2.4 Determination of Moisture-Vapor Permeability

The intrinsic moisture-vaporproofing of the materials submitted was desired rather than the permeability of a coating of these materials on paper as applied under an arbitrary set of conditions.

The measurement of moisture-vapor permeability by the usual gravimetric method with films of appreciable thickness would be very slow for those materials having moisture-vapor permeabilities sufficiently low to be of interest. Accordingly a dynamic method was developed, using an electric hygrometer.

40.2.5 Stability at Application Temperature

A test period of seven days at 212 ± 2 F was adopted for the study of the thermal stability of dipping compositions.

40.2.6 General Observations

The importance of odors and toxicity was recognized; quantitative measurements were planned. Samples known to contain materials which would impart odor or toxicity were eliminated.

40.2.7 Fungus Resistance

Three different organisms were studied, namely, *Chaetomium globosum*, *Metarrhizium glutinosum*, and *Aspergillus ustus*. The culture method of test was used and the relative toxicity of thirty-two different materials recorded.

40.3 SUMMARY

A survey of available materials, suitable for use as hot melts at temperatures not over 220 F, for improving the packaging of ration cartons was completed.

The need for methods of evaluation of fundamental properties was obvious from previous experience. Six properties were selected for evaluation, including viscosity-temperature relations, blocking characteristics, low-temperature flexibility, water-vapor permeability, stability at application temperatures, and general observations on odor and taste, and methods were developed for their evaluation. New techniques for low-temperature flexibility and water-vapor permeability of the coating material (as distinguished from coatings prepared under arbitrary conditions) were adapted from other fields and developed to a point necessary for comparison of the submitted materials.

The low-temperature flexibility test measured the extent to which a film of material will stretch before failure when loaded at a high rate of speed and at low temperatures (comparable to dropping a case in cold weather).

The water-vapor permeability test was more sensitive than previous methods, detecting transfer of as little as 2×10^{-5} gram of moisture vapor through a sample 9 sq in. in area. The size of sample may be varied without seriously affecting the sensitivity of the test in grams per unit area, thus permitting large samples to be tested in order to get overall averages more quickly or permitting small samples to be tested when only such samples are available or convenient.

None of the 12 samples submitted appeared suitable for immediate use. Only two hot melts showed any improvement in low-temperature flexibility over the paraffin-microcrystalline wax mixtures. These two materials had poor blocking characteristics at 140 F and viscosities higher than the waxes. Three materials showed improvement in blocking; of these, one had a high viscosity and poor moisture-vapor resistance; the other two had poor low-temperature flexibility.

The evaluation of the compounding agents submitted was started. Polyethylene and polyisobutylene (of the molecular weights first submitted) raised the viscosity of paraffin considerably when used in amounts of 10 to 15 per cent but improved the low-temperature flexibility somewhat.

A method for evaluating resistance of fungi to various toxicants was developed and applied to various materials, using *Chaetomium globosum*, *Metarrhizium glutinosum*, and *Aspergillus ustus* as test organisms. Investigations were also started on the mechanism of fungus growth on or in wax-dipped cartons, and organisms inside the sealed carton were isolated.

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Chapter 41

STORAGE OF CHLORINATED LIME

41.1 SUMMARY

A STUDY WAS MADE of the amount of heat liberated during the decomposition of chlorinated lime. The heat of reaction between chlorinated lime and several added impurities as well as the effect of varying moisture content and the catalysis by iron and by ferric oxide were also investigated.

The relative instability of chlorinated lime as a function of moisture content, as measured by the loss of available chlorine, can be expressed as follows:

1.5% : 5.0% : 10.5% : : 1.0 : 5.2 : 72.

The amount of heat liberated during the decomposition of chlorinated lime, containing no added impurity, could not be determined by the methods employed.

The following substances were tested for reactivity with chlorinated lime. They were listed in order of increasing activity, as measured by the amount of heat liberated per gram of added impurity: a vinylite resin, a phenolic resin, iron, asphalt, oil, and paper pulp. The reactivity of paper pulp and of oil was greater in chlorinated lime having low moisture content (1.5 per cent) than in that having high moisture content (10.5 per cent). The reactivity of asphalt was lowered by decreasing moisture content.

The presence of iron or of ferric oxide will catalyze the reactions between chlorinated lime and all of the above substances. However, only a very slight effect was noted in samples containing asphalt, the vinylite resin, or the phenolic resin. Catalysis was marked in samples containing paper pulp. The catalysis of iron and ferric oxide showed no dependence on moisture content.

The temperatures at which the reactions between chlorinated lime and the various impurities became evident were as follows: In samples containing 10.5 per cent moisture, paper pulp 35 C (95 F), oil 40 C (104 F), phenolic resin 85 C (185 F), vinylite resin 85 C (185 F), iron 95 C (203 F), and ferric oxide, no reaction determinable; in samples containing 5.0 per cent moisture, no reaction was noted below 75 C (167 F); in samples containing 1.5 per cent moisture, paper pulp 80 C (176 F), oil 85 C (185 F), the phenolic resin, the vinylite resin, asphalt, iron, and ferric oxide, no reaction determinable.

41.2 INTRODUCTION

This investigation was undertaken to extend the knowledge of the behavior of chlorinated lime during storage and to determine the factors responsible for the exothermic decomposition. The materials investigated were of the type known as Chamber Bleach, which is characterized by moisture content of 10 to 14 per cent, and Mechanical Bleach containing 5 per cent moisture or less.

The hazardous properties of chlorinated lime may be caused by two general properties of the material. It is an oxidizing agent, and as such, will react with substances susceptible to oxidation, with the liberation of heat during the process. Oxidizable substances may become associated with chlorinated lime by their being present in the materials from which the lime is manufactured, by introduction during manufacture, or because the chlorinated lime is stored in containers composed of reactive material. Common storage containers are iron drums and asphalt lined, iron ended, paper containers. All these container materials are susceptible to oxidation. Oil, an oxidizable substance, possibly could be introduced from manufacturing or packaging machinery. In addition to being an oxidizing agent, chlorinated lime is unstable. It undergoes measurable decomposition at room temperature, with the liberation of chlorine and oxygen. It is entirely possible that either of these two processes, i.e., reaction with oxidizable materials or spontaneous decomposition, may be catalyzed by impurities normally associated with chlorinated lime.

41.3 EXPERIMENTAL PROBLEMS

The experimental problems involved were to devise methods of determining the heat of decomposition of chlorinated lime, the heat of reaction of mixtures of chlorinated lime and oxidizable impurities, the relative rate of the various reactions and the temperatures at which the rates of the reactions became appreciable. The following factors were investigated:

1. Condition and composition of various chlorinated lime samples as received from Quartermaster depots.
2. The effect of moisture content on the decomposition of uncontaminated chlorinated lime.
3. The effect of the presence of oxidizable impurities and varying moisture content on the decomposition.
4. The heat evolved by the reaction of chlorinated lime with various added impurities.
5. The catalytic effect of iron and of ferric oxide on the decomposition of chlorinated lime in the presence of oxidizable impurities.

41.4 DISCUSSION OF RESULTS

The literature contained no reports of quantitative measurements on the amount of heat liberated during the decomposition of chlorinated lime. Contradictory evidence was presented regarding the extent and rate of decomposition of the material. Nevertheless, the weight of evidence indicated that chlorinated lime may be expected either to lose available chlorine at the rate of from 0.5 to 2.0 per cent per month which appeared to result in no serious evolution of heat, or to decompose violently within from 24 hours to several months after packaging, in which case the material would be a serious fire hazard. The 0.5 to 2.0 per cent per month loss of available chlorine appeared to be the normal rate of decomposition, which represented the inherent instability of the material, under normal storage conditions. The violent decomposition has been attributed to causes which include differences in manufacturing procedure, contamination by substances which act to catalyze the decomposition, and reaction with substances susceptible to oxidation.

Several instances have been recorded in which the stability of chlorinated lime has been stated to be inversely related to the moisture content of the material. The first phase of this investigation was an attempt to verify those observations, and further, to determine the effect of varying moisture content on the heat liberated during decomposition. The experiments indicated that the loss of available chlorine was inversely related to the moisture content. It should be realized that each of the samples was heated to 100 C (212 F), maintained at that temperature for the same length of time, and sampled for analysis after cooling. The magnitude of the loss on heating should not be associated with loss under actual storage conditions. It indicated the relative stability under conditions of the test. Should chlorinated lime behave in storage as under the conditions of the tests, the relationship between moisture content and loss of available chlorine would be as follows:

$$1.5\% : 5.0\% : 11.0\% :: 1.0 : 5.2 : 72.$$

However, despite the variation in stability with moisture content no evolution of heat could be observed during the decomposition. No attempt was made to trace the course of decomposition, so that a comparison with the various mechanisms of decomposition reported in the literature was not possible.

It was established that chlorinated lime, uncontaminated by appreciable amounts of oxidizable materials, could not be responsible for the liberation of sufficient heat to be a serious fire hazard. Therefore, it seemed pertinent that the reactions between chlorinated lime and the materials of construction for storage containers as well as substances likely to be found as contaminants, be investigated. The experiments described were designed to establish the resistance of paper, iron, asphalt, oil, ferric oxide, and two synthetic resins to oxidation (or chlorination) by chlorinated lime and to determine the effect of moisture content on the several reactions.

The results of these experiments were not quantitatively applicable to actual storage con-

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ditions. The reaction between chlorinated lime and any solid component will depend on the surface of the substances in contact. No a priori statement can be made concerning the distribution of contaminants in the chlorinated lime as packaged. The test samples were prepared so that, so far as possible, the extent of surface of the materials in the mixtures was constant. In all samples tested the contaminant was evenly distributed throughout the mixture. When oil was the contaminant, the components were mixed by grinding. The conditions in the test samples represented the greatest area of contact between the chlorinated lime and the impurity for the state of subdivision of the components and therefore represented the optimum conditions for the reactions.

The activity of the various impurities may be examined from two points of view. It may be reasoned that a substance is inactive if it does not react at an appreciable rate below a given temperature. This will be an important consideration in determining the maximum temperature allowed for storage, should a given impurity be present. Or, a substance may be considered inactive if the heat of reaction is low.

From the point of view of the temperature at which reaction with the various impurities becomes evident, moisture content is an important factor. Paper pulp will react at an appreciable rate with chlorinated lime containing 10.5 per cent moisture at a temperature as low as 35 C (95 F), whereas if the moisture content is lowered to 5.0 per cent, reaction is not evident at 75 C (167 F), and in the presence of 1.5 per cent moisture reaction is not appreciable at 80 C (176 F). The reaction with oil, the next most active substance, becomes evident at 40 C (104 F), 75 C (167 F), and 85 C (185 F) in chlorinated lime containing 10.5, 5.0, and 1.5 per cent moisture, respectively. The reaction of the two synthetic resins, and asphalt with chlorinated lime containing 10.5 per cent moisture could not be observed until heated to 85 C (185 F), while in the presence of only 1.5 per cent moisture no exothermic reaction was apparent up to 100 C (212 F). Iron presents a special problem. The rate of reaction with chlorinated lime is slow and little heat is evolved in samples containing 10.5, 5.0, and 1.5 per cent moisture, at temperatures up to 100 C (212 F). However, iron and ferric oxide are active catalysts for the reactions between chlorinated lime and paper pulp, oil, and to a lesser degree with asphalt.

The two synthetic resins were generally less active than the other substances tested. In the presence of 10.5 per cent moisture, reaction was not evident below 85 C (185 F) and no reactions were observed when the moisture was reduced to 1.5 per cent. Furthermore, neither iron nor ferric oxide had an appreciable catalytic effect on the reaction of the resins with chlorinated lime.

41.5 HYGROSCOPICITY AND RELATIONSHIP OF *p*H TO STABILITY

A method for the determination of the hygroscopicity of chlorinated lime and calcium hypochlorite was developed. Standard laboratory equipment was employed so that industrial laboratories could utilize the method without unnecessary delay. The equipment consisted of a constant temperature oven, glass desiccator, saturated KCl solution and weighing bottles. The results of the investigation showed that, in general, samples of chloride of lime were 33 to 100 per cent more hygroscopic than calcium hypochlorite.

The relationship of the *p*H of the samples to their stability as measured by the loss of available chlorine on heating was investigated. Samples were adulterated with 5, 10, 15, and 20 per cent of each of the following substances: quartz, calcium hydroxide, and boric acid. The quartz served as the inert diluent for comparison measurements. The *p*H of the samples were not appreciably increased by the addition of 5 to 20 per cent calcium hydroxide. The addition of 5 per cent boric acid caused very little change in the *p*H; 10, 15, and 20 per cent boric acid affected the *p*H of the calcium hypochlorite to a greater extent than the chlorinated lime.

The addition of varying quantitites of calcium hydroxide to samples of calcium hypochlorite did not appreciably increase their

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stability. Samples of chloride of lime were decidedly stabilized to the loss of available chlorine on heating. The addition of boric acid had a marked detrimental effect on the stability; the decrease in stability was caused by the presence of the boric acid rather than the effect of the boric acid on the *p*H.

41.6 GENERAL SPECIFICATIONS

This investigation suggested the following general specifications:

1. Samples exposed to 90 per cent relative humidity at 35 C for 24 hr should not gain more than 85 per cent by weight for calcium hypochlorite and 100 per cent by weight for chloride of lime.
2. The *p*H of the products should be between 11.0 and 12.0.
3. The moisture content should not be greater than 5 per cent for chloride of lime and calcium hypochlorite products.
4. The lime used for manufacture should be free from iron, cobalt, and manganese. Contamination by organic substances such as oil and cellulose should be prohibited.
5. The stability of the sample to the loss of available chlorine on heating should not be greater than 15 per cent for either the chloride of lime or the calcium hypochlorite.

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4. *Sealing of Navy Primers and Fuzes*, W. F. Singleton and W. C. Johnson, OSRD 6440, OEMsr-796, Service Project NO-288, E. I. duPont de Nemours and Co., Inc., Dec. 29, 1945. Div. 11-206.7-M2

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1. *Methods for the Purification of Water After Its Transportation and Storage in Cans, Drums and Tanks That Have Been Employed as Containers for Leaded Gasoline*, Robert A. Kehoe and J. Cholak, OSRD 1139, OEMsr-341, Service Project CE-19, University of Cincinnati, Dec. 9, 1942. Div. 11-203.72-M1

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Chapter 31

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1. *Solid Fuels for Heating Combat Rations* (Summary Report for the period from Aug. 9, 1943, to Feb. 29, 1944), Charles Paul McClelland and Robert Hayward Nimmo, Service Project QMC-26, Mellon Institute of Industrial Research, March 1944. Div. 11-208.42-M1

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1. *Shrinkproofing of Wool Knitted Items and Fabrics*, Vol. 1 (Final Report for the period from Mar. 1, 1944, through Dec. 31, 1944), Arthur L. Smith, Lydia R. Hornstein, and others, OEMsr-1055, Service Project QMC-34, National Bureau of Standards, January 1945. Div. 11-208.13-M1

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10. *Evaluation of Plastic Materials and Development of Production Testing* (Final Report for the period from Jan. 1, 1944, to June 30, 1944), R. W. Auxier and K. L. Landon, OEMsr-1055, Service Project QMC-30-C, Westinghouse Electric and Manufacturing Co., July 1944.

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OSRD APPOINTEES

DIVISION 11

Division 11 was organized on December 9, 1942 when former Division B of the NDRC was broken up into four new Divisions, 8, 9, 10, and 11, known as the Chemical Divisions. Former Division B was under the Chairmanship of Roger Adams and had ten sections, each of which had one or more subsections. Division 11 was made up of Sections B-7, B-8, part of B-9, and B-10 (together with subsections B-7-b, B-7-d, B-7-e, B-8-a, B-8-b, B-8-c, B-8-d, B-8-e, B-8-f, B-9-a, and B-9-d) of former Division B. Subsections B-9-b and B-9-c of Section B-9 were later incorporated in a new Division 19.

The list which appears below therefore shows essentially the organization since December 9, 1942. Although many changes were made during the years 1943-1945, the names of all appointees who held appointments to Division 11 at any time during this period have been included. In addition the names of men who held appointments in the sections and subsections of former Division B, but who did not have appointments to Division 11 following the reorganization, have been included so as to give a complete picture of the organization since the beginning of the work under NDRC.

Section 11.2 comprises Subsections B-8-a, B-8-b, B-8-c, B-8-d, B-8-e, B-8-f, B-9-a, and B-9-d of former Division B.

Chiefs

R. P. Russell

E. P. Stevenson

H. M. Chadwell

Technical Aide

D. Churchill, Jr.

Members

D. Churchill, Jr.
E. R. Gilliland
H. C. Hottel
H. F. Johnstone
W. K. Lewis
J. H. Rushton
R. P. Russell
T. K. Sherwood
E. P. Stevenson

SECTION 2

Chiefs

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CONTRACT NUMBERS, CONTRACTORS AND SUBJECT OF CONTRACTS

Contract Number	*Name and Address of Contractor*	*Subject*
NDCrc-3 (11-20)	Ethyl Gasoline Corporation, Detroit, Michigan	Oil and Gasoline Sabotage
NDCrc-15 (11-23)	University of Wisconsin, Madison, Wisconsin	Drying of Gases
NDCrc-29 (11-22)	Princeton University, Princeton, New Jersey	Paint Removers
NDCrc-37 (11-29)	Monsanto Chemical Company, Dayton, Ohio	Gasoline Sabotage
NDCrc-42 (11-27)	Bakelite Corporation, Bloomfield, New Jersey	Protective Coatings
NDCrc-50 (11-28)	University of Illinois, Urbana, Illinois	Removal of Salts from Sea Water
NDCrc-69 (11-48)	Harvard University, Cambridge, Massachusetts	Oxygen Breathing Equipment
NDCrc-92 (11-68)	Wesleyan University, Middletown, Connecticut	Flash Powders for Aerial Night Photography
NDCrc-165 (11-85)	Pennsylvania State College, State College, Pennsylvania	Hydraulic Fluids
OEMsr-6 (11-108)	Massachusetts Institute of Technology, Cambridge, Massachusetts	Evaporation of Drops
OEMsr-23 (11-115)	Harvard University, Cambridge, Massachusetts	Oxygen Breathing Equipment
OEMsr-52 (11-97)	Bakelite Corporation, Bloomfield, New Jersey	Protective Coatings
OEMsr-82 (11-149)	Arthur D. Little, Inc., Cambridge, Massachusetts	Investigation of the Use of Substitute Materials in the Manufacture of Cork Plugs Used in Naval Ordnance
OEMsr-104 (11-170)	Columbia University, New York City	Canister Design Calculations
OEMsr-130 (11-154)	Rockefeller Institute, New York City	Vesicant Thickening
OEMsr-158 (11-179)	Wesleyan University, Middletown, Connecticut	Underwater Flares
OEMsr-186 (11-152)	Pennsylvania State College, State College, Pennsylvania	Hydraulic Fluids
OEMsr-189 (11-209)	Rice Institute, Houston, Texas	Sabotage of Lubricating Oils
OEMsr-196 (11-190)	Harvard University, Cambridge, Massachusetts	Oxygen Breathing Equipment
OEMsr-204 (11-223)	Harvard University, Cambridge, Massachusetts	Report on Anti-fouling Paints

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Contract Number	*Name and Address of Contractor*	*Subject*
OEMsr-211 (11-219)	Bakelite Corporation, Bloomfield, New Jersey	Protective Coatings
OEMsr-231 (11-169)	Ethyl Gasoline Corporation, Detroit, Michigan	Oil and Gasoline Sabotage
OEMsr-283 (11-230)	Resinous Products, Philadelphia, Pennsylvania	Potable Water from Sea Water
OEMsr-306 (11-240)	Harvard University, Cambridge, Massachusetts	Data on Instantaneous Rates of Flow During Inspiration and Expiration
OEMsr-320 (11-242)	Harvard University, Cambridge, Massachusetts	Oxygen Breathing Equipment
OEMsr-321 (11-260)	Wesleyan University, Middletown, Connecticut	Development of Photoflash Bombs and Underwater Flares
OEMsr-341 (11-247)	University of Cincinnati, Cincinnati, Ohio	Methods of Purification of Water After its Transportation and Storage in Cans, Drums, and Tanks that have been Employed as Containers for Leaded Gasoline
OEMsr-347 (11-111)	Massachusetts Institute of Technology, Cambridge, Massachusetts	Canister Design
OEMsr-408 (11-271)	Pennsylvania State College, State College, Pennsylvania	Hydraulic and Recoil Fluids
OEMsr-425 (11-273)	Mine Safety Appliances Company, Pittsburgh, Pennsylvania	Development of Methods of Protecting Gasoline Tanks, Wing Spaces, and Cockpits Against Hazard of Explosive Mixtures of Gasoline and Air
OEMsr-428 (11-283)	Ethyl Corporation, Detroit, Michigan	Preliminary Engine Studies of Methods of Sabotage of Lubricating Oils
OEMsr-446 (11-218)	Bakelite Corporation, Bloomfield, New Jersey	Mechanical Methods of Cleaning Ship Bottoms
OEMsr-506 (11-343)	E. I. du Pont de Nemours and Company, Wilmington, Delaware	Investigation of Methods of Removal of Dust from Air Supplied to Aircraft Engines
OEMsr-565 (11-301)	Catholic University, Washington, D. C.	Preliminary Investigation of Possibilities of Increasing the Capacity of Lead Storage Batteries by Modification of the Negative Plate

CONTRACT NUMBERS, CONTRACTORS AND SUBJECT OF CONTRACTS
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Contract Number	*Name and Address of Contractor*	*Subject*
OEMsr-606 (11-299)	E. I. du Pont de Nemours and Company, Wilmington, Delaware	Experimental Studies of the Effect of Physical Properties of Viscous Fluids on the Break-up of Liquid Jets and Drops When Contacted by Air Stream
OEMsr-672 (11-342)	University of California, Berkeley, California	Investigation of the Removal of Oil from Harbor Waters by Means of Chemically Treated Sand
OEMsr-743 (11-359)	E. I. du Pont de Nemours and Company, Wilmington, Delaware	Development of Vesicant Thickeners
OEMsr-796 (11-377)	E. I. du Pont de Nemours and Company, Philadelphia, Pennsylvania	Linings for Fuel, Munitions, and Lubricant Containers; and Sealants for Navy Fuzes
OEMsr-820 (11-390)	Harvard University, Cambridge, Massachusetts	Study of Methods of Anaerobic Conversion of Urea to Ammonia and Aerobic Conversion of Ammonia to Nitrates
OEMsr-844 (11-410)	Universal Oil Products Company, Riverside, Illinois	Pilot Plant Studies of the Purification of Levinstein Mustard
OEMsr-873 (11-391)	Monsanto Chemical Company, Everett, Massachusetts	Design of Equipment for Producing Nitric Acid in China from Dilute Ammonia Solutions
OEMsr-896 (11-419)	The Texas Company, New York City	Sabotage of Automotive Equipment
OEMsr-897 (11-409)	The Texas Company, New York City	Pilot Plant Studies of the Purification of Levinstein Mustard by Solvent Extraction and Flash Distillation
OEMsr-928 (11-437)	E. I. du Pont de Nemours and Company, Wilmington, Delaware	Study of Sodium Hydride-Aluminum Mixtures Suitable for Field Generation of Hydrogen
OEMsr-930 (11-436)	National Research Corporation, Boston, Massachusetts	Development of Nonelectrolytic Method of Producing Lithium Hydride from Ores
OEMsr-946 (11-446)	Continental Can Co., Inc., New York City	Development of Hydrogen Generators for the Use of Sodium Hydride-Aluminum Mixtures
OEMsr-953 (11-431)	E. I. du Pont de Nemours and Company, Wilmington, Delaware	Evaluation of Processes for Producing Sodium Borohydride
OEMsr-1017 (11-421)	Massachusetts Institute of Technology, Cambridge, Massachusetts	Purification of Mustard

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OEMsr-1047 (11-462)	Arthur D. Little, Inc., Cambridge, Massachusetts	Development of Distilling Units for the Manufacture of Potable Water from Sea Water
OEMsr-1164 (11-471)	Massachusetts Institute of Technology, Cambridge, Massachusetts	Development of Solar Still
OEMsr-1169 (11-473)	Massachusetts Institute of Technology, Cambridge, Massachusetts	Gas Generator for Pressurizing Flame Throwers. More Rapid Inflation of Life Rafts in Cold Waters
OEMsr-1194 (11-476)	University of Indiana, Bloomington, Indiana	Investigation of Methods of Producing Magnesium Fluoride
OEMsr-1206 (11-479)	General Printing Ink Corporation, New York City	Plane Crash Dye Markers
OEMsr-1223 (11-474)	Comstock and Wescott, Inc., Niagara Falls, New York	Marine Type Electrocoating for Gasoline Containers
OEMsr-1244 (11-478)	Curtiss-Wright Corporation, Buffalo, New York	Protection of Airplane Fuel Tanks from Explosion
OEMsr-1259 (11-485)	Rutgers University, New Brunswick, New Jersey	Effect of Marine Organisms on Corrosion and Paint Degradation
OEMsr-1271 (11-482)	Rice Institute for the Advancement of Literature, Science and Art, Houston, Texas	Suppression of Dust by Ground Treatment
OEMsr-1354 (11-502)	Grinnell Corporation, Providence, Rhode Island	Gas Generator for Pressurizing Flame Throwers
OEMsr-1373 (11-506)	General Printing Ink Corporation, New York City	Advance Positions Identification
OEMsr-1395 (11-507)	E. I. du Pont de Nemours and Company, Wilmington, Delaware	Exterior Ballistics of Liquid Filled Shell
OEMsr-1452 (11-510)	University of Illinois, Urbana, Illinois	Concentrated Hydrogen Peroxide
OEMsr-1453 (11-509)	Massachusetts Institute of Technology, Cambridge, Massachusetts	Concentrated Hydrogen Peroxide
OEMsr-1465 (11-513)	Massachusetts Institute of Technology, Cambridge, Massachusetts	Investigation of Preparation of Dilute Hydrogen Peroxide by Application of Fluid Catalyst Technique

CONTRACTS ON QUARTERMASTER PROBLEMS

OEMsr-718 (11-375)	University of Cincinnati, Cincinnati, Ohio	Studies and Investigations in Connection with the Improvement of Wearing Qualities of Leather for Military Uses
OEMsr-854 (11-415)	Ethyl Corporation, Detroit, Michigan	Deleading of Gasoline

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Contract Number	*Name and Address of Contractor*	*Subject*
OEMsr-862 (11-411)	Textile Foundation, National Bureau of Standards, Washington, D. C.	Water Repellent Treatment
OEMsr-888 (11-418)	Massachusetts Institute of Technology, Cambridge, Massachusetts	Rot and Termite Prevention
OEMsr-929 (11-430)	Massachusetts Institute of Technology, Cambridge, Massachusetts	Troop Feeding Program
OEMsr-1014 (11-450)	Smithsonian Institution, Washington, D. C.	Military Ski Poles, Western Hemisphere Bamboo, Sources for
OEMsr-1055 (11-500)	National Academy of Sciences, Washington, D. C.	QMC Problems
OEMsr-1055 (11-500) AN-20 (sub-contract)	Mellon Institute of Industrial Research, Pittsburgh, Pennsylvania	Development of Suitable Buoyant Material for Use in Life Vests, etc.
OEMsr-1055 (11-500) QMC-7 (sub-contract)	Worcester Polytechnic Institute, Worcester, Massachusetts	Leather Sole; Substitute for
OEMsr-1055 (11-500) QMC-17 (sub-contract)	University of Cincinnati, Cincinnati, Ohio	Studies and Investigations in Connection with the Improvement of Wearing Qualities of Leather for Military Uses
OEMsr-1055 (11-500) QMC-19A (sub-contract)	University of Chicago, Chicago, Illinois	Deleading of Gasoline
OEMsr-1055 (11-500) QMC-20 (sub-contract)	Textile Foundation, Bureau of Standards, Washington, D. C.	Water Repellent Treatment
OEMsr-1055 (11-500) QMC-22 (sub-contract)	Massachusetts Institute of Technology, Cambridge, Massachusetts	Rot and Termite Prevention
OEMsr-1055 (11-500) QMC-23 (sub-contract)	Massachusetts Institute of Technology, Cambridge, Massachusetts	Troop Feeding Program
OEMsr-1055 (11-500) QMC-24 (sub-contract)	Smithsonian Institution, Washington, D. C.	Military Ski Poles, Western Hemisphere Bamboo, Sources for
OEMsr-1055 (11-500) QMC-27 (sub-contract)	Columbia University, New York City	Flameproofing
OEMsr-1055 (11-500) QMC-29 (sub-contract)	Textile Foundation, National Bureau of Standards, Washington, D. C.	Impregnated Clothing
OEMsr-1055 (11-500) QMC-30A (sub-contract)	Rensselaer Polytechnic Institute, Troy, New York	Doron
OEMsr-1055 (11-500) QMC-30B (sub-contract)	Arthur D. Little, Inc., Cambridge, Massachusetts	Doron
OEMsr-1055 (11-500) QMC-30C (sub-contract)	Westinghouse Electric Mfg. Co., East Pittsburgh, Pennsylvania	Doron
OEMsr-1055 (11-500) QMC-30D (sub-contract)	General Electric Company, Schenectady, New York	Doron

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Contract Number	*Name and Address of Contractor*	*Subject*
OEMsr-1055 (11-500) QMC-30F (sub-contract)	Monsanto Chemical Company, Dayton, Ohio	Doron
OEMsr-1055 (11-500) QMC-30G (sub-contract)	American Cyanamid Company, 1937 West Main St., Stamford, Connecticut	Doron
OEMsr-1055 (11-500) QMC-30I (sub-contract)	Plaskon Division of Libby Owens Ford Company	Doron
OEMsr-1055 (11-500) QMC-31 (sub-contract)	Temple University, Philadelphia, Pennsylvania	Organic Coatings
OEMsr-1055 (11-500) QMC-32 (sub-contract)	Brooklyn Polytechnic Institute, Brooklyn, New York	Thin Films from Synthetic Elastomers
OEMsr-1055 (11-500) QMC-33 (sub-contract)	Massachusetts Institute of Technology, Cambridge, Massachusetts	Wear Resistance of Apparel Textiles
OEMsr-1055 (11-500) QMC-34 (sub-contract)	Textile Foundation, National Bureau of Standards, Washington, D. C.	Shrinkproofing of Wool Knit Items and Fabrics
OEMsr-1055 (11-500) QMC-36 (sub-contract)	Brooklyn Polytechnic Institute, Brooklyn, New York	Improvement of Coated Fabrics

SERVICE PROJECTS

The projects listed below were transmitted to the Executive Secretary, NDRC, from the War or Navy Department through either the War Department Liaison Office for NDRC or the Office of Research and Inventions (formerly the Coordinator of Research and Development), Navy Department.

Service Project Number	*Title*
	Army Projects
AC-2	Research on Fuels, Fuel Additives, and Lubricant Additives.
AC-7	Constant Viscosity Hydraulic Fluid.
AC-8	Hydraulic Packing Material.
AC-29	Miscellaneous Research on Photographic Equipment.
AC-38	Development of Oxygen Mask Embodying the Principle of Full Head Coverage and Equipped with Antifog Goggles.
AC-93	Data on Instantaneous Rates of Flow During Inspiration and Expiration.
CE-19	Determination of a Suitable and Practicable Method for Cleaning Gasoline Containers to Make Them Safe for Holding Drinking Water.
CWS-1	Aerosols—Their Generation, Stabilization and Precipitation.
CWS-4	Methods of Preparation of Certain Non-arsenical Organic Compounds.
CWS-5	Test of Pro-knock Materials: Special Problems Relating to Internal Combustion Engines.
CWS-7	Fundamental Study of Gas Mask Absorbents.
CWS-10	Gas Generator for Pressurizing Flame Throwers.
CWS-12	Materials for Thickening and Increasing the Viscosity of Vesicants.
CWS-13	Catalyst for Prevention of Corrosion of Steel Containers by Liquid Vesicants.
CWS-15	Improved Filter Materials.
CWS-16	Improved Filter Design.
CWS-21	Study of Incendiary Materials.
CWS-31	Exterior Ballistics of Liquid-Filled Shell.
OD-61	Development of Ideal Recoil Oil.
OD-130	Anti-Corrosive Linings for Gasoline Containers.
OD-154	Suppression of Dust of Flash Around Field Guns.
QMC-17	Investigation of Leathers, tanning methods and special treatment to leather for the purpose of making leathers most adaptable to military use and increasing its serviceability.
QMC-19	Deleading of Gasoline.
QMC-20	Water Repellency Treatments: evaluation procedures for
QMC-22	Insect Damage to QM Equipment; survey of Present Knowledge of Dangers and Prevention.
QMC-23	Troop Feeding Program, a Critique and Compilation of Available Information.

SERVICE PROJECTS
(Continued)

Service Project Number	*Title*
	Army Projects
QMC-24	Substance for Bamboo for Military Purposes.
QMC-28	Marine Electroplating Process.
SC-44	Improved Hydrogen Generation Methods.
SOS-12	Development of Material for Marking Foliage for Identification of Position by Planes.
	Navy Projects
NA-105	Protection of Aircraft Fuel System Against Gunfire.
NA-122	Improvement of Organic Protective Coatings for Use on Naval Aircraft.
NE-100	Plane Crash Dye Marker.
NE-102	Design of Rubber Life Raft; extension to include more rapid inflation of life rafts.
NL-B1(i)	Problems Related to the Manufacturing and Use of Potassium Peroxide in Oxygen Breathing Equipment.
NL-B2	Development of Non-toxic and Non-inflammable Paint Removers.
NL-B4	Protective Coatings.
NL-B5	Methods of Rendering Sea Water Potable.
NL-B36	Study of Spreading of Droplets of All Types of Mustard Gas and Lewisite on Cloth and on Metal and Painted Surfaces.
NO-B37	Substitute for Cork in Ordnance Plugs.
NO-126	Study of Suitable Containers for Various Types of Corrosive Chemical and Smoke-Producing Agents.
NO-189	Development of Improved Method of Producing Pure Magnesium Fluoride.
NO-266	Investigation and Development of Petroleum and Non-Petroleum Hydraulic Fluids.
NO-288	Sealing Agent(s) for Projectile Base Fuzes.
NS-103	Removal of Oil or Gasoline Film from Surface of Water.
NS-110	Possible Improvements on Submarine Storage Batteries and Other Lead Storage Batteries.
NS-129	Development of Fireproof Paint.
NS-168	Development of Distilling Units for the Manufacture of Potable Water from Sea Water; extension to include Solar Energy-Type Stills.
NS-235	Research on the Effect of Micro-Organisms on Corrosion.
	Army-Navy Projects
AN-13	Development of Non-contaminating 55-gallon Drums for Petroleum Products.
	Neither Army nor Navy Projects
Chinese-1	Nitric Acid from Urea.
NACA-1	Removal of Dust from Air Intake of Aircraft.
SAC-9	Sabotage of Automotive Equipment.

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